



FINAL
COMPREHENSIVE QUALITY
ASSURANCE MANUALS
RCRA FACILITY INVESTIGATION
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DEPARTMENT OF THE NAVY SOUTHERN DIVISION NAVAL FACILITIES ENGINEERING COMMAND CHARLESTON, SOUTH CAROLINA

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VOLUME V Book 1 of 3



COMPREHENSIVE QUALITY ASSURANCE PLAN

Savannan Division

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COMPREHENSIVE QUALITY ASSURANCE PLAN

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3.0 STATEMENT OF POLICY

Savannah Laboratories is committed to providing quality data and will endeavor to use good quality control and quality assurance practices for all field sampling and laboratory analytical procedures in order to ensure the best possible precision, accuracy, and representativeness of results from testing of environmental samples.

The objectives of the QA program are to:

- (1) Properly collect, preserve, and store all samples;
- (2) Maintain adequate custody records from sample collection through reporting and archiving of results;
- (3) Use properly trained analysts to analyze all samples by approved methods and within holding times;
- (4) Produce QC verifiable data which can be documented to show that the system was calibrated and within precision and accuracy control limits;
- (5) Accurately calculate, check, and enter all data into the Laboratory Information Management System; and
- (6) Document all the above activities in order that all data can be independently validated.

Savannah Laboratories intends to follow all procedures referenced in this plan and to conform to EPA and state regulatory agency guidelines for each project reported. Any changes in EPA or other regulatory procedures will be incorporated during periodic revisions of this plan.

Adherence to the procedures of this plan is assured by the assignment of an experienced project manager to each project. The project manager coordinates and is responsible for all phases of Savannah Laboratories' involvement in the project, including pre-project planning, sample bottle preparation, field sampling, computer entry of work, approving analytical and quality control data, final review of report, and discussion of results with client. The project managers are assisted by QA managers and staff at each laboratory.

The QA Plan will be utilized by all five Savannah Laboratories facilities. Additionally, all labs use identical Standard Operating Procedures (SOP), all data are incorporated into a single Laboratory Information Management System (LIMS) network which generates common QA limits, etc., and is accessible to all employees. Each project is directed by a single project manager who supervises all employees involved on the project, and also reviews, approves, and signs all data reports.

The following sections of this QA plan detail the organizational structures and procedures through which all laboratory results are generated.

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4.0 ORGANIZATION AND RESPONSIBILITY

Savannah Laboratories and Environmental Services, Inc. has laboratory facilities in, and conducts field operations from, Savannah, Georgia; Tallahassee, Florida; Mobile, Alabama; Deerfield Beach, Florida; and Tampa, Florida. All five facilities are structured under a common administrative, data management, and quality assurance (QA) system as outlined in Figures 4.1, 4.2, 4.3, 4.4, 4.5, and 4.6.

Duties of the key personnel are as follows:

- A) Company President
 - Establish corporate policy;
 - Plan and oversee laboratory infrastructure construction/acquisition;
 - Negotiate contractual agreements; and
 - 4) Other administrative and budgetary functions.
- B) Company Vice President
 - Provide guidance to lab directors;
 - 2) Establish and maintain company-client relationships; and
 - 3) Assist president in establishing and carrying out corporate policy.
- C) Controller
 - 1) Supervise administration section;
 - Prepare financial reports;
 - 3) Coordinate risk management program; and
 - 4) Assist corporate officers with budgetary problems.
- D) Corporate Technical Staff
 - 1) Provide technical support for all divisions;
 - Coordinate technical activities affecting all divisions;
 - Write SOPs and other technical documents; and
 - 4) Inform all divisions about new methods.

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E) Business Manager

- Supervise accounting section;
- 2) Coordinate purchases and payables; and
- 3) Maintain equipment inventory and business records.

F) Laboratory Director

- 1) Responsible for day-to-day operation of lab;
- Provide project manager guidance;
- 3) Establish production priorities; and
- 4) Approve hiring decisions.

G) Project Manager

- 1) Initial contact with client on individual job tasks;
- 2) Prepare all work plans, schedules and manpower allocations;
- 3) Initiate all procurement for the projects;
- 4) Day-to-day supervision of the project team including analytical department managers, field sampling crews and data management personnel;
- 5) Coordinate financial and contractual aspects of the projects;
- 6) Provide formatting and technical review of all reports;
- Provide day-to-day communication with the client;
- 8) Exercise final review and approval on all reports and invoices for the project; and
- Respond to post project inquiries.

H) QA Manager

- Coordinate with the project manager, and laboratory manager in order to insure that project QA is maintained;
- Be available to discuss QA activities and results with client;
- Prepare QA reports to management;
- 4) Perform periodic system audits;

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- 5) Review not-in-compliance reports and approve corrective actions;
- 6) Coordinate the preparation and approval of all QA plans, method SOPs and QA audit responses; and
- 7) Coordinate and be present during all external QA Audits.

I) Laboratory Manager

- Coordinate all production activities;
- Work with project managers to ensure project objectives are met;
- 3) Provide guidance to department managers; and
- 4) Interview and hire technical personnel.

J) Sample/Data Manager

- 1) Schedule bottle orders and supervise bottle prep staff;
- Supervise custody staff;
- 3) Coordinate with project manager and field/sampling manager on scheduling field sampling efforts;
- 4) Identify and document custody discrepancies and communicate with client on custody problems; and
- 5) Supervise data management staff including computer login, data entry, report preparation, and data archiving personnel.

K) Field/Sampling Manager

- 1) Coordinate and schedule sampling crews;
- 2) Prepare sampling reports; and
- 3) Ensure sampling protocols are followed.

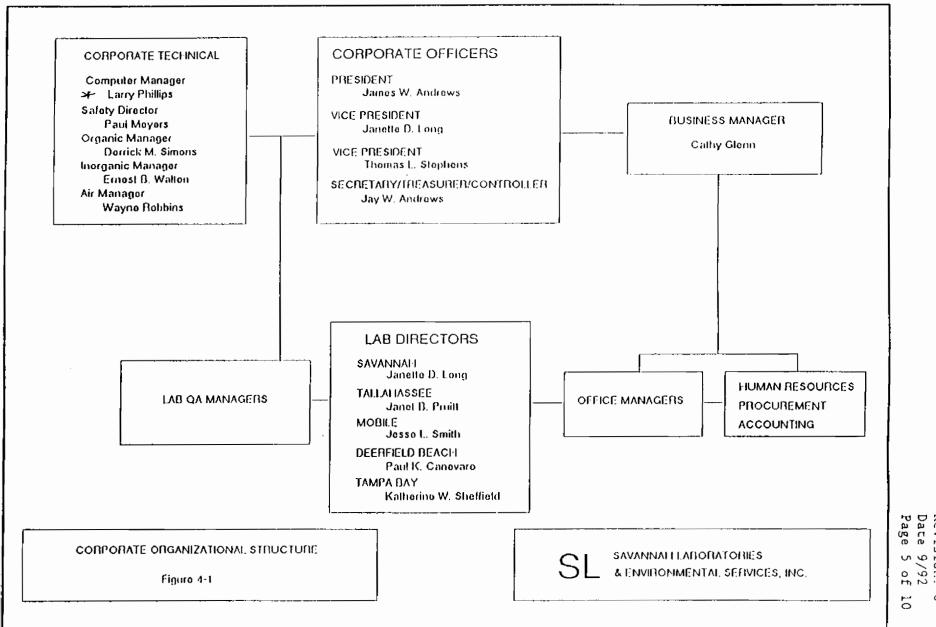
L) Department Manager

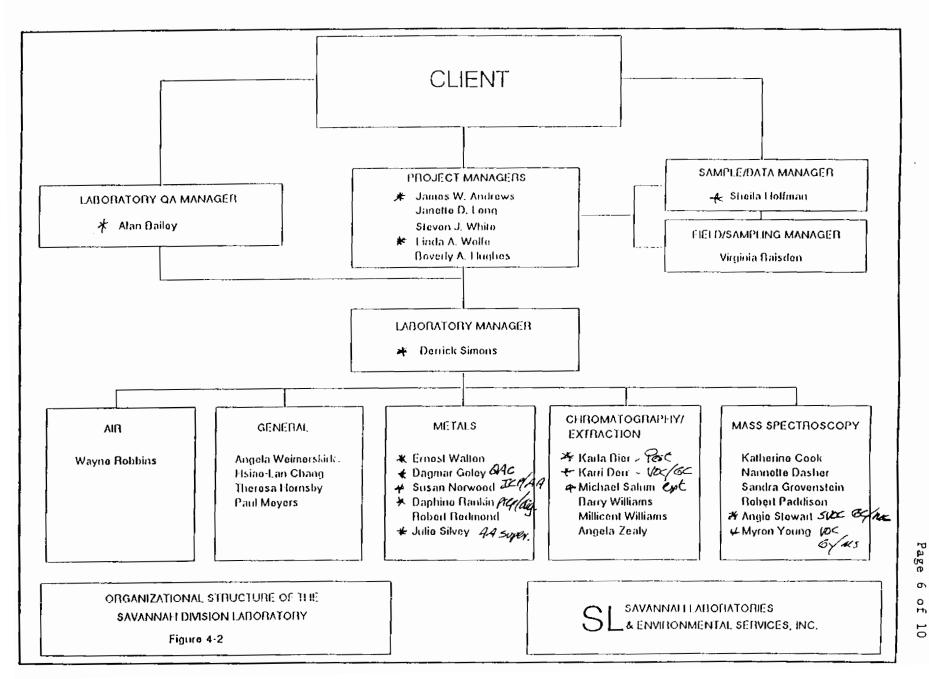
- Organize work flow in department;
- 2) Assure adequate inventory of reagents and equipment;
- 3) Ensure effective maintenance and repair of instrumentation;
- 4) Investigate and evaluate new methodology and equipment; and
- 5) Train new employees.

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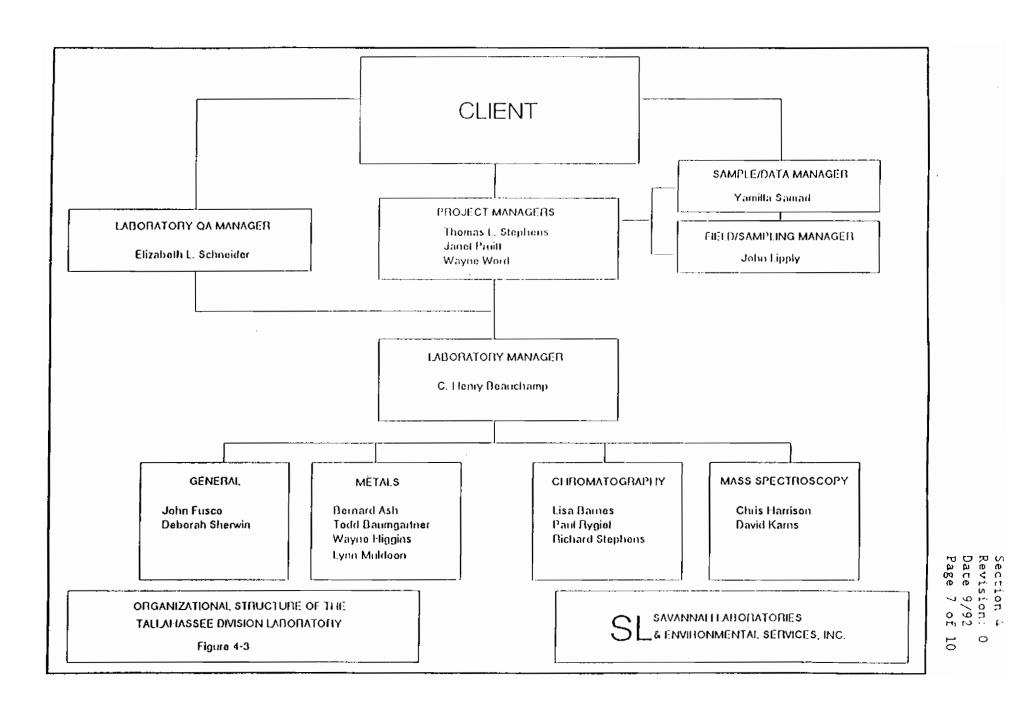
A list of all technical employees and resumes for each of the professionals in the organization are provided in Section 16.0.

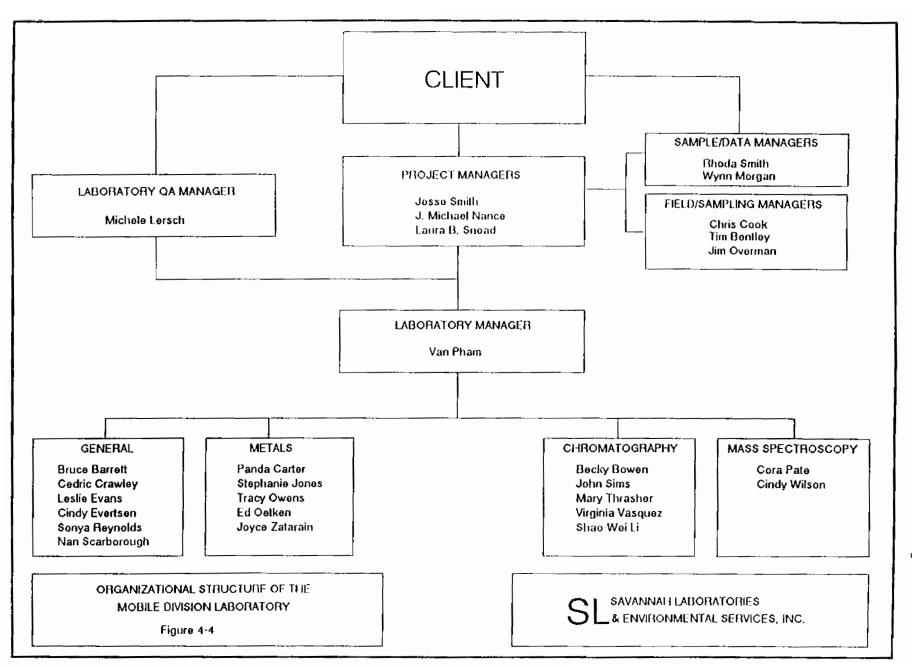
In case of instrument failure, high sample volume, or rapid turnaround requirements, samples are interchanged among the five facilities. In these situations, samples or preserved extracts are transported under EPA recommended chain-of-custody, handling and storage procedures. This inter-exchange of workload practice is possible because of single administrative structure, the use of identical analytical and QA protocols, and the fact that all five facilities are tied into (via telephone modem) a central computerized Laboratory Information Management System (LIMS).





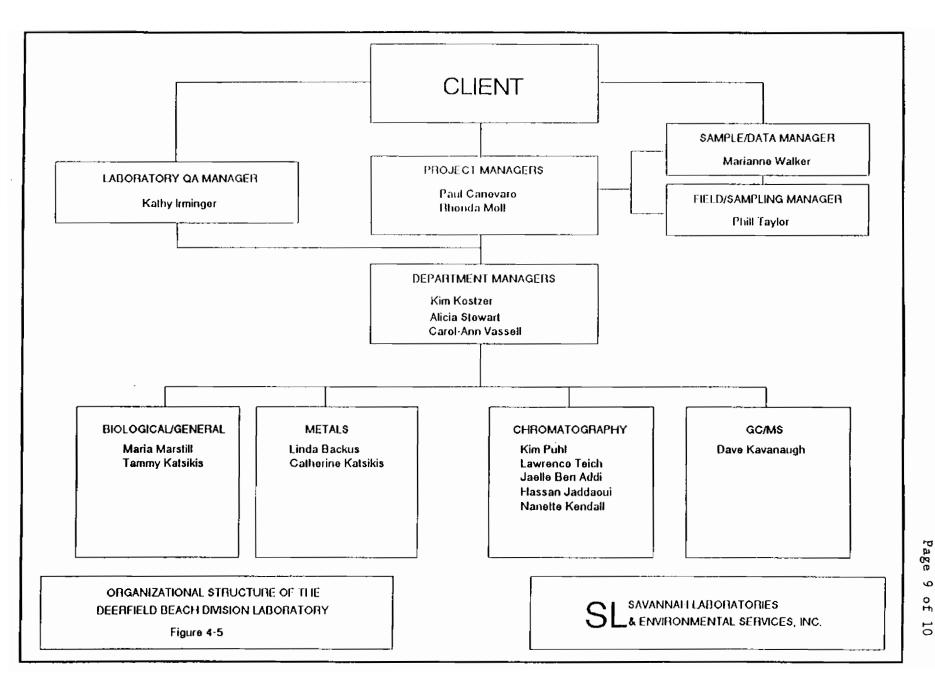
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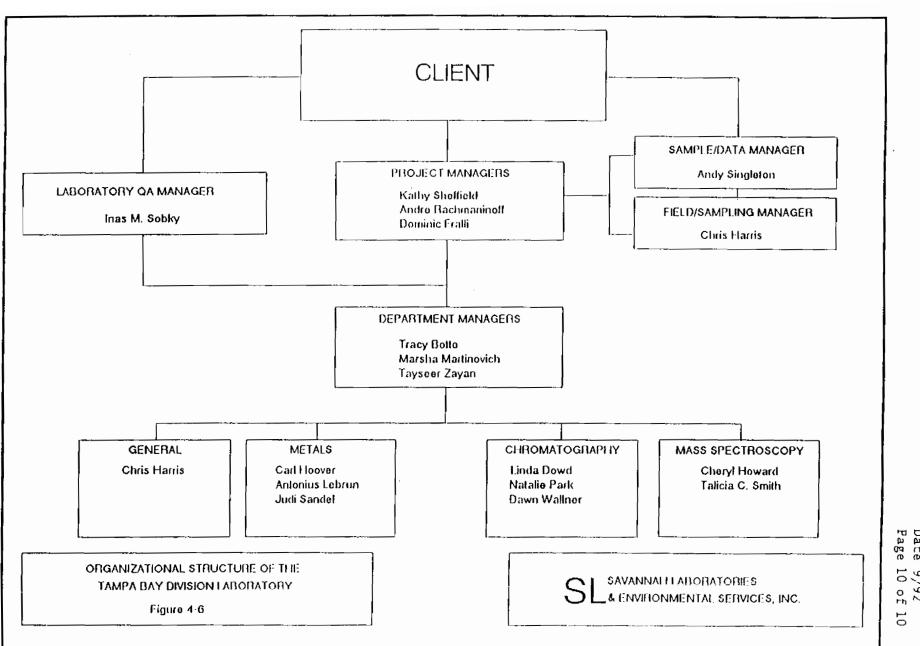


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5.0 QUALITY ASSURANCE OBJECTIVES (PRECISION, ACCURACY, AND PQLs)

Savannah Laboratories has a comprehensive quality assurance program which is based on the program outlined in EPA's Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80), in the Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA, 1979) and in the Association of Official Analytical Chemists' Quality Assurance Principles for Analytical Laboratories.

The key to Savannah Laboratories QA/QC program is strict adherence to the program during all phases of the project including: presampling discussions; sample collection, preservation, transportation and storage; sample login and tracking; laboratory analyses; and validation and reporting of results.

Project and QC data from all facilities are entered into a single Laboratory Information Management System (LIMS). The LIMS provides a computerized mechanism for storing field and login information, tracking sample holding times, scheduling and preparing laboratory work sheets, storing results and QC data, reviewing results and relating them to their corresponding QC data, and printing reports and invoices. The Project Manager, QA Manager, and data management and reporting personnel have direct access via a CRT terminal to all project and QA data from all five facilities.

Tables 5.1 and 5.2 list the laboratory parameters determined by Savannah Laboratories, the methodology, the QA objectives for precision, accuracy and the normal practical quantification limits (PQLs) for relatively clean environmental samples. Accuracy control limits are for lab control standards (LCS) or blank spike recoveries and do not apply to matrix spike (advisory only). Table 5.3 gives the same information for field parameters.

PRECISION

The Savannah Laboratories objective for precision is to meet the precision data generated by the applicable method validation on similar matrices. Relative percent difference (RPD) is used to express precision between two replicate values. In routine analyses, the values for most parameters are usually below PQLs; therefore, precision data are derived from duplicate matrix spike or lab control standard results.

The relative percent difference (RPD) is calculated as:

$$RPD = \frac{V1 - V2}{(V1 + V2)/2} \times 100$$

V1, V2 - The two values obtained by analyzing the duplicate samples.

ACCURACY

The Savannah Laboratories objective for accuracy is to meet the accuracy data generated by the applicable method validation on similar matrices. Percent recovery (XR) is used to express accuracy from the analysis of blank spikes and other QC samples.

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The percent recovery (%R) is calculated as below:

- SAV The background value, value obtained by analyzing the sample
- SA Concentration of the spike added to the sample
- SPV Value obtained by analyzing the sample with the spike added

COMPARABILITY

The Savannah Laboratories objective for comparability is to strive toward the comparability of sample parameters on similar matrices as they relate to precision and accuracy determinations. Strict adherence to QA/QC procedures promotes the comparability of one set of reference data to another or comparability of data among all facilities.

REPRESENTATIVENESS

The Savannah Laboratories objective for representativeness of field samples is to ensure that a set of data accurately depicts the distinguishing characteristic of a sample source. Representativeness is enhanced by an attempt to mix samples prior to aliquot removal. Results are considered reliable and representative if the sample distribution is within statistically defined bounds of the population mean and variance.

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Atuminum	200.7A	65	75-125	0-20	20
	200.7/6010(3010)	3/2	75-125	0-20	200
	Saltwater	5	50-140	0-40	10
	CLP	45	80-120	0-20	200
Antimony	200.7/6010(3010***)	3/2	75-125	0-20	50
	204.2/7041(3005)	3/2	75-125	0-20	20
	CLP	45	80-120	0-20	60
Arsenic	200.7A 200.7/6010(3010) 206.2/7060(3020***) 206.3/7061 206.3/7061-Saltwater CLP	65 3/2 3/2 3/2 3/2/5 45	75-125 75-125 75-125 60-140 60-140 80-120	0-20 0-20 0-20 0-40 0-40 0-20	10 100 10 2.0 0.10
Barium	200.7A	65	75-125	0-20	1.0
	200.7/6010(3010)	3/2	75-125	0-20	10
	CLP	45	75-125	0-20	200
Beryllium	200.7/6010(3010)	3/2	75-125	0-20	5.0
	210.2/7091(3020)	3/2	75-125	0-20	5.0
	CLP	45	80-120	0-20	5.0
Boron	200.7/6010(3010***)	3/2	<i>7</i> 5-125	0-20	50
Cadmium	200.7A	65	75-125	0-20	0.50
	200.7/6010(3010)	3/2	75-125	0-20	5.0
	213.2/7131(3020)	3/2	75-125	0-20	1.0
	Saltwater	5	60-140	0-40	0.050
	CLP	45	80-120	0-20	5.0
Catcium	200.7/6010(3010)	3/2	75-125	0-20	500
	CLP	45	80-120	0-20	5000
Chromium	200.7A	65	75-125	0-20	1.0
	200.7/6010(3010)	3/2	75-125	0-20	10
	218.2/7191(3020)	3/2	75-125	0-20	10
	CLP	45	80-120	0-20	10
Chromium, hexavalent	7196	2	75-125	0-20	10
Cobalt	200.7/6010(3010)	3/2	75-125	0-20	10
	CLP	45	80-120	0-20	50
Copper	200.7A 200.7/6010(3010) 220.1/220.2(3020) Saltwater CLP	65 3/2 3 5 45	75-125 75-125 75-125 60-140 80-120	0-20 0-20 0-20 0-20 0-40 0-20	2.5 25 10 0.50 25
Iron	200.7A 200.7/6010(3010) 236.2(3020) Saltwater CLP	65 3/2 3 5 45	75-125 75-125 75-125 60-140 80-120	0-20 0-20 0-20 0-20 0-40 0-20	5.0 50 10 2.0
Lead	200.7A	65	75-125	0-20	5.0
	200.7/6010(3010)	3/2	75-125	0-20	50
	239.2/7421(3020)	3/2	75-125	0-20	5.0
	Saltwater	5	60-140	0-40	0.50
	CLP	45	80-120	0-20	3.0
Lithium	3500-Li B	4	75-125	0-20	100
Magnesium	200.7/6010(3010)	3/2	75-125	0-20	500
	CLP	45	80-120	0-20	5000
Manganese	200.7A	65	75-125	0-20	1.0
	200.7/6010(3010)	3/2	75-125	0-20	10
	CLP	45	80-120	0-20	15

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL=+ (Ug/L)
Mercury	245.1/7470	3/2	75 - 125	0-20	0.20
	Saltwater	5	60-140	0-40	0.10
	CLP	45	80-120	0-20	0.20
Molybdenum	200.7/6010(3010)	4/2	75-125	0-20	10
Nickel	200.7A	65	75 - 125	0-20	4.0
	200.7/6010(3010)	3/2	75 - 125	0-20	40
	249.2 Saltwater	3 5	75-125	0-20	10
	CLP	45	60-140 80-120	0-40	1.0
Phosphorus	200.7***/6010***(3010***)	3/2	75-125	0-20	50
Potassium	200.7/6010(3010)	3/2	75-125	0-20	1000
	258.1/7610(3010)	3/2	75-125	0-20	100
	CLP	45	80-120	0-20	5000
Selenium	200.7/6010(3010)	3/2	75 - 125	0-20	100
	270.2/7740(3020***)	3/2	75-125	0-20	10
	270.3/7741	3/2	60-140	0-40	2.0
	270.3/7741 - Saltwater	3/2/5	60-140 80-125	0-40	0.10
# 112 ·	CLP	45		0-20	5.0
Silica	200.7/6010(3010***)	3/2	75 - 125	0-30	500
Silver	200.7A	65	75 - 125	0-20	1.0
	200.7/6010(3010***) 272.1	3/2	75-125 75-125	0-20 0-20	10 10
	272.2/7761	3/2	75-125	0-20	1.0
	Saltwater	5	60-140	0-40	0.050
	CLP	45	80-120	0-20	10
Sodium	200.7/6010(3010)	3/2	75-125	0-20	500
	273.1	3	75-125	0-20	500
	CLP	45	80-120	0-20	5000
Strontium	200.7***/6010***(3010***)	3/2	75-125	0-20	10
Thallium	200.7/6010(3010)	3/2	75 - 125	0-20	500
	279.2/7841(3020)	3/2	75 - 125	0-20	10
	CLP	45	80-120	0-20	<u>! 10</u>
Tin	200.7***V/6010***V	3/2	75-125	0-20	50
	(3010***V) 1 282.2	3	75-12 5	0-20	50
Titaniមា	200.7***/6010***(3010***)	3/2	75-125	0-20	10
Tributyl tin	Atomic absorption	40	60-140	0-40	0.0040
Vanadium	200.7/6010(3010)	3/2	75-125	0-20	10
	CLP	45	80-120	0-20	50_
Zinc	200.7A	65	75-125	0-20	2.0
	200.7/6010(3010)	3/2	75-125	0-20	20
	Saltwater	5	60-140	0-40	1.0
	CLP	45	80-120	0-20	20
Zinc phosphide	FDER Special Method	31	10-210	0-80	2.0
Zirconium	200.7***/6010***(3010***)	2	75-125	0-20	5000

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PGL** (mg/L)
Acidity	305.1/402	3/4	75-125	0-30	10
Alkalinity	310.1/403	3/4	75-125	0-30	1.0
Ammonia (as N)	350.1 350.3	3 3	90-110 75-125	0-30 0-30	0.030 0.050
Ammonia, un-ionized	FL-DER	60	NA	NA	0.010
Bicarbonate	403	4	NA	NA NA	1.0
BOO	405.1/507	3/4	60-140	0-30	2.0
Bromate	300.0	3	75-125	0-30	1.0
8romide	9056/300.0 320.1	2/3 3	75 - 125 75 - 125	0-30 0-30	1.0
Carbon, total organic	415.1/9060	3/2	60-140	0-40	1.0
Carbonate	403	4	МА	NA	1.0
CB00	507	4	NA .	0-30	2.0
Chloride	325.2 325.3/9252 407A 9056/300.0	3 3/2 4 2/3	85-115 75-125 75-125 75-125	0-30 0-30 0-30 0-30	1.0 1.0 1.0 1.0
Chlorine, residual	408A 330.4 330.5	4 3 3	NA NA NA	0-30 0-30 0-30	1.0 1.0 1.0
Chlorophyll	1002G	4	NA.	0-30	0.00010
coo	508B 410.2 410.4	4 3 3	60-140 60-140 60-140	0-30 0-30 0-30	20 20 20
Coliform, fecal, MPN	908C	4	NA	NA	2 MPN/ 100 mL
Coliform, fecal, MF	909C	4	NA.	ŅĀ	1 col/100 m
Coliform, total, MPN	908A	4	NA	HA	2 MPN/100 m
Coliform, total, HF	909A	4	AK	NA	1 col/100 m
Color	110.2/204A	3/4	NA :	0-40	5 PCU
Corrosivity	203	4	NA	NA	NA
Cyanate	412K	4	60-140	0-40	0.10
Cyanide, amenable to chlorination	9012 335.1/9010	2 3/2	NA NA	0-50 0-40	0.010 0.01D
Cyanide, reactive	7.3.3.2	2	NA	0-50	0.010
Cyanide, total	335.3/9012 335.2/9010 CLP	3/2 3/2 45	85-115 75-125 85-115	0-30 0-30 0-30	0.010 0.010 0.010
Cyanide, weak and dissociable	412H	4	NA	0-40	0.010
Fluoride	340.2	3	75-125	0-30	0.20

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD	REFERENCE	ACCURACY*	PRECISION*	PQL**
Formaldehyde	(Prep)	35	(% Rec) 70-125	(% RPD) 0-30	(mg/L)
					0.25
Malogens, total organic	450.1/9020	3/2	60-140	0-40	0.010
Hardness, total	314A	4	NA NA	<u>NA</u>	3.3
Hydrogen ion (pH)	150.1/9040	3/2	90-110	0-10	NA
Nitrate (as N)	353.2 9056/300.0	3 2/3	85-115 75-125	0-30 0-30	0.050
	352.1	3	75-125	0-30	0.10
	353.3	3	75-125	0-30	0.050
Nitrate-Nitrite (as N)	353.2	3	85-115	0-30	0.050
Ignitability	1010	2	NA_	NA NA	NA NA
Nitrite (as N)	353.2 354.1	3 3	85-115 75-125	0-30 0-30	0.050
	9056/300.0	2/3	75-125	0-30	0.050
	353.3	3	75-125	0-30	0.050
Nitrogen, total Kjeldahl (TKN)	351.2	3 3	65-135	0-40	0.10
	351.3		75-125	0-30	0.10
Nitrogen, organic	EPA-CE	46	NA .	NA	0.10
Nitrogen, total	EPA-CE	46	NA NA	NA	0.15
Odor	140.1/207	3/4	NA.	NA NA	1 TON
Oil & Grease	413.1/503A 413.2/5038	3/4 3/4	60-140 60-140	0-30 0-30	5.0 1.0
Orthophosphate (as P)	365.1	3	80-120	0-30	0.050
	365.2 365.3	3 3	75-125 75-125	0-30 0-30	0.050
	9056/300.0	2/3	75-125	0-30	0.10
Oxygen, dissolved	360.1	3_	NA	0-30	0.10
Petroleum hydrocarbons	418.1/503E	3/4	60-140	0-30	1.0
Phenolics, total recoverable	420.2/9066 420.1/9065	3/2 3/2	75-125 75-125	0-30 0-30	0.010
Phosphorus, organic (as P)	365.4	3	NA NA	NA NA	0.10
	365.4	3	60-140	0-40	0.10
Phosphorus, total (as P)	365.3	3	60-140	0-40	0.050
	365.2	3	60-140	0-40	0.10
Plate count, heterotrophic	907	4	AK	AK	1000 CFU/L
Radioactivity, alpha	900.0/9310/703	54/2/4	48-162	0-25	2.0 oCi/L
Radioactivity, beta	900.0/9310/703	54/2/4	NA	0-25	2.0 pCi/L
Residue, dissolved	160.1/209B	3/4	75-125	0-30	5.0
Residue, suspended	160.2/209C	3/4	75-125	0-30	5.0
Residue, total	160.3/209A	3/4	60-140	0-40	5.0
Residue, volatile	160.4/2090	3/4	NA	0-40	5.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (mg/L)
Salinity	210	4	NA.	NA	100
Settleable matter	160.5/209E	3/4	NA	0-40	0.20 mL
Silica, dissolved	370.1	3	75-125	0-30	10
Specific conductance	120.1/9050	3/2	90-110	0-10	1.0 umho/cm
Specific gravity	213E	3	NA	NA .	NA AK
Streptococcus, fecal, MPN	910A	4	NA	NA	2 MPN/100 mL
Streptococcus, fecal, MF	910B	4	NA.	NA	1 col/100 mL
Sulfate	9036 375.3 375.4 9056/300.0	2 3 3 2/3	80-120 75-125 75-125 75-125	0-30 0-30 0-30 0-30	5.0 5.0 5.0 5.0
Sulfide	376.2/427 9030-st	3/4	60-140 50-150	0-40 0-50	0.40
Sulfide, reactive	7.3.4.2	2	NA	0-50	0.40
Sulfite	428 377.1	4 3	75-125 75-125	0-30 0- 3 0	1.0
Surfactants (MBAS)	425.1	3	70-130	0-30	0.10
Temperature	170.1	3	, MW	0-10	NA
Thiocyanate	412.L	4	60-140	0-40	0.10
THM formation potential	5710	4	NA NA	NA	0.010
Turbidity	180.1/214A	3/4	60-140	0-30	0.10 NTU

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY*	PRECISION*	PQL** (ug/L)
Bromodichloromethane	501.1/501.2	49/50	54-128	0-40	0.50
Bromoform	501.1/501.2	49/50	50-140	0-40	0.50
Chlorodibromomethane	501.1/501.2	49/50	60-125	0-40	0.50
Chloroform (MS)	501.1/501.2	49/50	65-137	0-40	0.50
Bromodichloromethane	501.3	30	50-125	0-40	0.50
8romoform_	501.3	30	50-127	0-40	0.50
Chlorodibromomethane	501.3	30	50-125	0-40	0.50
Chloroform (MS)	501.3	30	50-125	0-40	0.50
Surrogate - Bromochloromethane ,	501.1/501.2/501.3	49/50/30	46-118	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD	REFERENCE	ACCURACY*	PRECISION*	PQL**
<u> </u>	(Prep)		(% Rec)	(% RPD)	(ug/L)
Bromobenzene	502.1	51	57-129	0-40	0.50
Bromochloromethane	502.1	51	60-125	0-40	0.50
Bromodichloromethane	502,1	51	55-125	0-40	0.50
Bromeform	502.1	51	50-140	0-40	5.0
Bromomethane	502.1	51	60-140	0-40	5.0
Carbon tetrachloride	502.1	51	55-125	0-40	0.50
Chlorobenzene (MS)	502.1	51	25-134	0-29	0.50
Chloroethane	502.1	51	39-147	0-40	1.0
Chloroform	502.1	51	35-131	0-40	0.50
Chloromethane	502.1	51	54-125	0-40	1.0
2-Chlorotoluene	502.1	51	58-125	0-40	0.50
4-Chlorotoluene	502.1	51	50-140	0-40	0.50
Dibromochloromethane	502.1	51	55-140	0-40	0.50
1,2-Dibromoethane	502.1	51	54-132	0-40	1.0
Dibromomethane	502.1	51	50-140	0-40	0.50
1,2-Dichlorobenzene	502.1	51	56-134	0-40	0.50
1,3-0ichlorobenzene	502.1	51	58-125	0-40	0.50
1,4-Dichlorobenzene	502.1	51	51-129	0-40	0.50
Dichlorodifluoromethane	502.1	51	43-163	0-40	1.0
1,1-Dichloroethane	502.1	51	60-125	0-40	0.50
1,2-Dichloroethane	502.1	51	59-131	0-40	0.50
1,1-Dichloroethene (MS)	502.1	51	55-133	0-29	0.50
cis-1,2-Dichloroethene	502.1	51	55-125	0-40	0.50
trans-1,2-Dichloroethene	502.1	51	60-125	0-40	0.50
1,2-Dichloropropane	502.1	51	53-125	0-40	0.50
1,3-Dichloropropane	502.1	51	55-125	0-40	0.50
2,2-Dichloropropane	502.1	51	50-150	0-40	0.50
1,1-Dichloropropene	502.1	51	61-125	0-40	0.50
cis-1,3-Dichloropropene	502.1	51	51-129	0-40	0.50
trans-1,3-Dichloropropene	502.1	51	54-125	0-40	0.50
Methylene chloride	502.1	51	49-125	0-40	
1,1,1,2-Tetrachloroethane	502.1	51			0.50
1,1,2,2-Tetrachloroethane	502.1	51	59-125 58-125	0-40	0.50

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION® (% RPD)	PQL** (Ug/L)
Tetrachloroethene	502.1	51	50-125	0-40	0.50
1,1,1-Trichloroethane	502.1	51	55-125	0-40	0.50
1,1,2-Trichloroethane	502.1	51	57-125	0-40	0.50
Trichloroethene (MS)	502.1	51	51-142	0-24	0.50
Trichtoroftuoromethane	502.1	51	55-125	0-40	0.50
1,2,3-Trichtoropropane	502.1	51	59-130	0-40	1.0
Vinyl chloride	502.1	51	55-155	0-40	1.0
Surrogate - Bromochloromethane	502.1	51	46-118	AK	АК

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Acetone	502.2***	51	50-130	0-40	25
Benzene (MS)	502.2	51	73-144	0-22	0.50
Bromobenzene	502.2	51	57-129	0-40	0.50
Bromochloromethane	502.2	51	60-125	0-40	0.50
Bromodichloromethane	502.2	51	55-125	0-40	0.50
Bromoform	502.2	51	50-140	0-40	1.0
Bromomethane	502.2	51	60-140	0-40	1.0
n-Butylbenzene	502.2	51	55-125	0-40	0.50
sec-Butylbenzene	502.2	51	55-125	0-40	0.50
tert-Butylbenzene	502.2	51	55-125	0-40	0.50
Carbon tetrachloride	502.2	51	55-125	0-40	0.50
Chiorobenzene (MS)	502.2	51	25-134	0-29	0.50
Chloroethane	502.2	51	39-147	0-50	1.0
Chloroform	502.2	51	35-131	0-40	0.50
Chloromethane	502.2	51	54-125	0-40	1.0
2-Chlorotoluene	502.2	51	58-125	0-40	0.50
4-Chlorotoluene	502.2	51	50-140	0-40	0.50
Dibromochloromethane	502.2	51	55-140	0-40	0.50
1,2-Dibromo-3-chloropropane	502 .2	51	57-129	0-40	5.0
1,2-Dibromoethane	502.2	51	54-132	0-40	1.0
Dibromomethane	502.2	51	50-140	0-40	0.50
1,2-Dichlorobenzene	502.2	51	56-134	0-40	0.50
1,3-Dichlorobenzene	502.2	51	58-125	0-40	0.50
1,4-Dichlorobenzene	502.2	51	51-129	0-40	0.50
Dichlorodifluoromethane	502.2	51	43-163	0-50	1.0
1,1-Dichloroethane	502.2	51	60-125	0-40	0.50
1,2-Dichloroethane	502.2	51	59-131	0-40	0.50
1,1-Dichloroethene (MS)	502.2	51	55-133	0-29	0.50
cis-1,2-Dichloroethene	502.2	51	55-125	0-40	0.50
trans-1,2-Dichloroethene	502.2	51	60-125	0-40	0.50
1,2-Dichloropropane	502.2	51	53-125	0-40	0.50
1,3-Dichloropropane	502.2	51	55-125	0-40	0.50
2,2-Dichloropropane	502.2	51	50-150	0-40	0.50
1,1-Dichloropropene	502.2	51	61-125	0-40	0.50
cis-1,3-Dichloropropene	502.2	51	51-129	0-40	0.50
trans-1,3-Dichloropropene	502.2	- 51	54-125	0-40	0.50
Ethylbenzene	502.2	51	55-125	0-40	0.50
Hexachlorobutadiene	502.2	51	55-125	0-40	0.50
Isopropylbenzene	502.2	51	55-125	0-40	0.50
4-Isopropyltoluene	502.2	51	55-125	0-40	0.50

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Methylene chloride	502.2	51	49-125	0-40	1.0
Methyl ethyl ketone	502.2***	51	60-130	0-40	10
4-Methyl-2-pentanone	502.2***	51	65-125	0-40	10
Naphthalene	502.2	51	55-125	0-40	0.50
Propylbenzene	502.2	51	55-125	0-40	0.50
Styrene	502.2	51	55-125	0-40	0.50
1,1,1,2-Tetrachloroethane	502.2	51	59-125	0-40	0.50
1,1,2,2-Tetrachloroethane	502.2	51	58-125	0-40	1.0
Tetrachloroethene	502.2	51	50-125	0-40	0.50
Toluene (MS)	502.2	51	68-138	0-17	0.50
1,2,3-Trichlorobenzene	502.2	51	55-125	0-40	0.50
1,2,4-Trichlorobenzene	502.2	51	55-125	0-40	0.50
1,1,1-Trichloroethane	502.2	51	55-125	0-40	0.50
1,1,2-Trichloroethane	502.2	51	57-125	0-40	0.50
Trichloroethene (MS)	502.2	51	51-142	0-24	0.50
Trichlorofluoromethane	502.2	51	55-125	0-40	0.50
1,2,3-Trichloropropane	502.2	51	59-130	0-40	1.0
1,2,4-Trimethylbenzene	. 502.2	51	55-125	0-40	0.50
1,3,5-Trimethylbenzene	502.2	51	55-125	0-40	0.50
Vinyl chloride	502.2	51	55-155	0-40	1.0
o-Xylene	502.2	51	55-125	0-40	0.50
m-Xylene	502.2	51	55-125	0-40	0.50
p-Xylene	502.2	51	55-125	0-40	0.50
Surrogate - 2-Bromo-1-chloropropane	502.2	51	81-113	NA	НA
Surrogate - Fluorobenzene	502.2	51	69-108	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPO)	PQL** (Ug/L)
Benzene (MS)	503.1	51	73-144	0-22	1.0
p-Bromofluorobenzene	503.1***	51	58-125	0-40	1.0
Bromobenzene	503.1	51	55-125	0-40	1.0
n-Butylbenzene	503.1	51	30-126	0-40	1.0
sec-Butylbenzene	503.1	51	47-125	0-40	1.0
2,3-Benzofuran	503.1***	<u>5</u> 1	35-125	0-40	1.0
tert-Butylbenzene	503.1	51	51-125	0-40	1.0
Chlorobenzene (MS)	503.1	51	25-134	0-29	1.0
o-Chlarotoluene	503.1	51	55-125	0-40	1.0
p-Chlorotoluene	503.1	51	58-125	0-40	1.0
p-Cymene	503.1***	51	57-125	0-40	1.0
Cyclopropylbenzene	503.1***	51	60-125	0-40	1.0
p-Dichlorobenzene	503.1	51	63-127	0-40	1.0
m-Dichlorobenzene	503.1	51	65-125	0-40	1.0
o-Dichlorobenzene	503.1	51	58-125	0-40	1.0
Ethylbenzene	503.1	51	66-125	0-40	1.0
Hexachlorobutadiene	503.1	51	23-125	0-40	1.0
Isopropylbenzene	503.1	51	61-125	0-40	1.0
Naphthalene	503.1	51	57-135	0-40	1.0
n-Propylbenzene	503.1	51	56-125	0-40	1.0
Styrene	503.1	51	50-125	0-40	1.0
Toluene (MS)	503.1	51	68-138	0-17	1.0
Trichtoroethene	503.1	51	68-125	0-40	1.0
Trichlorotoluene	503.1***	51	56-125	0-40	1.0
Tetrachloroethene	503.1	51	60-128	0-40	1.0
1,3,5-Trimethylbenzene	503.1	51	52-125	0-40	1.0
1,2,4-Trimethylbenzene	503.1	51	48-125	0-40	1.0
1,2,4-Trichlorobenzene	503.1	51	55-127	0-40	1.0
1,2,3-Trichlorobenzene	503.1	51	52-125	0-40	1.0
p-Xylene	503.1	51	58-125	0-40	1.0
m-Xylene	503.1	51	56-125	0-40	1.0
o-Xylene	503.1	51	59-125	0-40	1.0
Surrogate - a,a,a-Trifluorotoluene	503.1	51	77-140	NA	AK

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Chloropicrin	504***V	51	60-140	0-40	0.010
1,2-Dibromoethane (MS) (EDB)	504 8011	51 2	60-140 60-140	0-40 0-40	0.020 0.020
1,2-0ibromo-3-chloropropane (MS)	504 8011	51 2	60-140 60-140	0-40 0-40	0.020 0.020
1,1-Dichloropropane	504***V	51	60-140	0-40	2.0
1,3-Dichloropropene	504***V	51	60-140	0-40	1.0
Methyl isothiocyanate	504***V	51	60-140	0-40	20

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL**
Alachlor	505	51	48-156	0-30	1.0
Aldrin	505/508	51	48-124	0-30	0.010
Atrazine	505	51	20-159	0-40	1.0
alpha BHC	508	51	52-132	0-30	0.010
beta BHC	508	51	55-135	0-30	0.020
delta BHC	508	51	57-147	0-30	0.010
gamma BHC (Lindane) (MS)	505/508	51	52-136	0-18	0.010
alpha Chlordane	505/508	51	51-147	0-30	0.010
gamma Chlordane	505/508	51	51-147	0-30	0.010
technical Chlordane	505	51	31-141	0-40	0.10
Chloroneb	508	51	51-143	0-30	0.50
Chlorobenzilate	508	51	58-148	0-30	0.20
Chlorothalonil	508	51	51-131	0-30	0.20
Dacthal (DCPA)	508	51	53-153	0-30	0.20
4,41-000	508	51	57-157	0-30	0.020
4,41-DDE	508	51	51-147	0-30	0.020
4,4'-DDT (MS)	508	51	67-137	0-28	0.050
Dieldrin (MS)	505/508	51	51-143	0-46	0.020
Endosulfan I	508	51	47-127	0-30	0.020
Endosulfan II	508	51	52-132	0-30	0.050
Endosulfan sulfate	508	51	41-163	0-40	0.10
Endrin (MS)	505/508	51	57-142	0-23	0.020
Endrin aldehyde	508	51	48-128	0-30	0.10
Etridiazole	508	51	58-143	0-30	0.10
Meptachlor (MS)	505/508	5	42-129	0-22	0.010
Meptachlor epoxide	505/508	51	47-127	0-30	0.020
Hexachlorobenzene	505/508	51	34-164	0-40	0.050
Hexachlorocyclopentadiene	505	51	26-120	0-40	0.20
Methoxychlor	505/508	51	37-163	0-40	0.50
cis-Nonachlor	505	51	49-171	0-30	0.050
trans-Nonachlor	505	51	47-133	0-30	0.020
cis-Permethrin	508	51_	51-131	0-30	1.0
trans-Permethrin	508	51	61-151	0-30	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Propachlor	508	51	58-131	0-30	1.0
Simazine	505	51	56-132	0-40	1.0
Toxaphene	505/508	51	60-168	0-40	1.0
Trifluralin	508	51_	58-119	0-30	0.050
PCB 1016	505/508	51	50-130	0-40	0.50
PCB 1221	505/508	51	50-130	0-40	0.50
PCB 1232	505/508	<u>5</u> 1	50-123	0-40_	0.50
PCB 1242	505/508	51	50-130	0-40	0.50
PCB 1248	505/508	51	50-130	0-40	0.50
PCB 1254	505/508	51	28-148	0-40	0.50
PCB 1260	505/508	51	28-148	0-40	0.50
Surrogate - Dibutylchlorendate (DBC)	505/508	51	28-151	NA NA	МА
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	505/508	51	22-126	NA.	NA
Surrogate - Decachlorobiphenyl (DCB)	505/508	51	25-126	NA NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Alachior (MS)	507	51	45-140	0-30	1.0
Ametryn	507	51	51-131	0-30	1.0
Atraton	507	51	47-135	0-30	5.0
Atrazine (MS)	507	51	40-125	0-30	1.0
Bromacil	507	51	55-127	0-30	2.0
Butachlor	507	51	56-136	0-30	1.0
Butylate	507	51	38-145	0-76	2.0
Carboxin	507	51	62-142	0-30	1.0
Chlorpropham	507	51	49-137	0-30	1.0
Cycloate	507	51	46-159	0-47	2.0
Demeton	507***	51	50-140	0-30	2.5
Diazinon (MS)	507	51	40-140	0-30	1.0
Dichlorvos	507	51	57-137	0-30	1.0
Diphenamid	507	51	53-133	0-30	2.0
Disulfoton	. 507	51	10-178	0-60	2.0
EPTC	507	51	46-154	0-55	2.0
Ethoprop	507	51	55-120	0-30	2.5
Fenamiphos	507	51	58-122	0-30	1.0
Fenarimot	507	51	59-179	0-30	1.0
Fluridone	507	51	51-123	0-30	5.0
Hexazinone (MS)	507	51	50-130	0-30	1.0
Merphos	507	51	56-136	0-30	2.5
Metalaxyl	507***	51	40-160	0-30	1.0
Metolachlor	507	51	53 - 133	0-30	1.0
Metribuzin	507	51	61-141	0-30	1.0
Mevinphos	507	51	51-139	0-30	10
MGK 264	507	51	50-150	0-30	20
Molinate (MS)	507	51	37-127	0-74	2.0
Napropamide	507	51	61-141	0-30	1.0
Norflurazon	507	51	54-134	0-30	1.0
Parathion, ethyl (MS)	507***	51	18-171	0-28	1.0
Parathion, methyl	507***	51	50-135	0-30	1.0
Pebulate	507	51	58-130	0-30	2.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Prometon	507	51_	42-114	0-30	1.0
Prometryn	507	51	51-125	0-30	1.0
Pronamide	507	51	51-131	0-30	2.0
Propazine	507	51	60-124	0-30	1.0
Simazine (MS)	507	51	60-140	0-30	1.0
Simetryn	507	51	59-139	0-30	1.0
Stirophos	507	51	58-138	0-30	10
Tebuthiuron	507	51	48-120	0-30	5.0
Terbacil	507	51	57-137	0-30	10
Terbufos	507	51	57-137	0-30	1.0
Terbutryn	507	51	58-130	0-30	1.0
Triademeton	507	51	61-125	0-30	1.0
Vernolate	507	51	53-133	0-30	2.0
Surrogate - Triphenylphosphate	507	51	40-125	NA	NA.

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Acifluorfen	515.1	51	21-185	9-40	1.0
Bentazon	515.1	51	32-182	0-40	1.0
Chloramben	515.1	51	53-169	0-40	1.0
2,4-D (MS)	515.1	51	25-129	0-60	0.50
Dalapon	515.1	51	40-160	0-50	10
2,4-08	515.1	51	48-126	0-40	0.50
Dicamba	515.1	51	40-144	0-40	0.50
3,5-Dichlorobenzoic acid	515.1	51	53-151	0-40	1.0
Dichlorprop	515.1	51	46-168	0-40	0.50
Dinoseb	515.1	51	49-129	0-40	0.50
5-Hydroxydicamba	515.1	51	53-153	0-40	1.0
4-Nitrophenol	515.1	51	25-229	0-40	1.0
Pentachlorophenol	515.1	51	36-224	0-40	1.0
Pictoram	515.1	51	44-138	0-40	0.50
2,4,5-1	515.1	51	25-145	0-62	0.50
2,4,5-TP (Silvex) (MS)	515.1	51	10-151	0-81	0.50

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Benzene (HS)	524.2	51	73-144	0-22	1.0
Bromobenzene	524.2	51	62-122	0-30	1.0
Bromochloromethane	524.2	51	50-150	0-40	1.0
Bromodichloromethane	524.2	51	49-151	0-40	1.0
Bromoform	524.2	51	60-130	0-30	1.0
Bromomethane	524.2	51	50-150	0-40	2.0
n-Butylbenzene	524.2	51	50-150	0-40	1.0
sec-Butylbenzene	524.2	51	50-150	0-40	1.0
tert-Butylbenzene	524.2	51	50-150	0-40	1.0
Carbon tetrachloride	524.2	51	55-121	0-40	1.0
Chlorobenzene (MS)	524.2	51	68-136	0-17	1.0
Chloroethane	524.2	51	50-150	0-40	2.0
Chloroform	524.2	51	63-133	0-30	1.0
Chloromethane	524.2	51	50-150	0-40	2.0
2-Chlorotoluene	524.2	51	50-150	0-40	1.0
4-Chlorotoluene	524.2	51	50-150	0-40	1.0
Dibromochloromethane	524.2	51	47-137	0-40	1.0
1,2-Dibromo-3-chloropropane	524.2	51	46-154	0-40	2.0
1,2-Dibromoethane	524.2	51	51-135	0-30	1.0
Dibromomethane	524.2	51	58-130	0-30	1.0
1,2-Dichlorobenzene	524.2	51	60-130	0-30	1.0
1,3-Dichlorobenzene	524.2	51	50-150	0-40	1.0
1,4-Dichtorobenzene	524.2	51	63-151	0-30	1.0
Dichlorodifluoromethane	524.2	51	60-132	0-30	1.0
1,1-Dichloroethane	524.2	51	65-135	0-30	1.0
1,2-Dichloroethane	524.2	51	57-127	0-30	1.0
1,1-Dichloroethene (MS)	524.2	51	60-136	0-19	1.0
cis-1,2-Dichloroethene	524. 2	51	50-150	0-40	1.0
trans-1,2-Dichloroethene	524.2	51	58-128	0-30	1.0
1,2-Dichloropropane	524.2	51	61-131	0-30	1.0
1,3-Dichloropropane	524.2	51	60-130	0-30	1.0
2,2-Dichloropropane	524.2	51	50-150	0-40	1.0
1,1-Dichloropropene	524.2	51	50-150	0-40	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
cis-1,3-Dichloropropene	524.2	51	50-150	0-40	1.0
trans-1,3-Dichloropropene	524.2	51	50-150	0-40	1.0
Ethylbenzene	524.2	51	50-150	0-40	1.0
Hexachlorobutadiene	524.2	51	50-150	0-40	1.0
Isopropylbenzene	524.2	51	50-150	0-40	1.0
4-Isopropyltoluene	524.2	51	50-150	0-40	1.0
Methylene chloride	524.2	51	40-160	0-50	1.0
Naphthalene	524.2	51	50-150	0-40	1.0
n-Propylbenzene	524.2	51	50-150	0-40	1.0
Styrene	524.2	51	50-150	0-30	1.0
1,1,1,2-Tetrachloroethane	524.2	51	50-150	0-40	1.0
1,1,2,2-Tetrachloroethane	524.2	51	62-150	0-30	1.0
Tetrachloroethene	524.2	51	60-126	0-30	1.0
Taluene (MS)	524.2	51	68-138	0-17	t.0
1,2,3-Trichlorobenzene	524.2	51	50-150	0-40	1.0
1,2,4-Trichlorobenzene	524.2	51	50-150	0-40	1.0
1,1,1-Trichloroethane	524.2	51	65-135	0-30	1.0
1,1,2-Trichloroethane	524.2	51	50-150	0-40	1.0
Trichloroethene (MS)	524.2	51	66-136	0-20	1.0
Trichlorofluoromethane	524.2	51	69-139	D-30	1.0
1,2,3-Trichloropropane	524.2	51	50-150	0-40	t.0
1,2,4-Trimethylbenzene	524.2	51	50-150	0-40	1.0
1,3,5-Trimethylbenzene	524.2	51	50-150	0-40	1.0
Vinyl chloride	524.2	51	34-131	0-30	1.0
o-Xyl ene	524.2	51	62-132	0-30	1.0
m-Xylene	524.2	51	50-150	0-40	1.0
p-Xylene	524.2	51	61-141	0-30	1.0
Surrogate - p-Bromofluorobenzene	524.2	51	79-125	NA	AK
Surrogate - 1,2-Dichlorobenzene-d4	524.2	51	77-135	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Acenaphthylene	525	51	49-131	0-40	0.50
Alachior	\$25	51	40-136	0-40	1.0
Aldrin (MS)	525	51	42-116	0-25	1.0
Anthracene	525	51	20-150	0-40	0.50
Atrazine (MS)	525	51	43-177	0-50	2.0
Benz(a)anthracene	525	51	30-150	0-50	0.50
Benzo(b)fluoranthene	525	51	20-128	0-40	0.50
Benzo(k)fluoranthene	525	51	30-150	0-40	0.50
Benzo(a)pyrene	525	51	20-160	0-40	0.50
Benzo(g,h,i)perylene	525	51	10-140	0-50	0.50
Butyl benzyl phthalate	525	51	<u>30-</u> 130	0-40	1.0
alpha Chlordane	525	51	43-167	0-40	1.0
gamma Chlordane	525	51	41-159	0-40	1.0
Chrysene	525	51	30-150	0-40	0.50
Dibenz(a,h)anthracene	525	51	10-110	0-50	0.50
Di-n-butyl phthalate	525	51	20-164	0-50	10
Diethylphthalate	525	51	22-180	0-50	2.0
bis(2-ethylhexyl)adipate	525	<u>5</u> 1	27-125	0-50	1.0
bis(2-ethylhexyl)phthalate	525	51_	28-142	0-50	2.0
Dimethylphthalate	525	51	20-150	0-40	1.0
Endrin	525	51	20-163	0-40	5.0
Fluorene	525	51	36-184	0-50	0.50
Mentachlor	525_	51	25-150	0-40	1.0
Heptachlor epoxide	525	51	30-158	0-40	1.0
Hexachlorobenzene	525	51	6-144	0-40	0.50
Hexachlorocyclopentadiene	525	51	9-140	0-50	0.50
Indeno(1,2,3-cd)pyrene	525	51	16-150	0-50	0.50
Lindane (MS)	525	51	52-136	0-18	1.0
Methoxychlor	525	51	6-182	0-50	1.0
trans-Nonachlor	525	51	45-125	0-40	1.0
Pentachlorophenol (MS)	525	51	15-139	0-39	3.0
Phenanthrene	525	51	45-138	0-40	0.50
Pyrene (MS)	525	51	36-153	0-21	0.50

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD	REFERENCE	ACCURACY*	PRECISION*	PQL**
	(Prep)		(% Rec)	(% RPD)	(ug/L)
Simazine	525	51	40-167	0-50	2.0
Toxaphene	525	51	20-142	0-50	50
PCBs:		·	· ·-		
2-Chlorobiphenyl	525	51	38-142	0-40	5.0
2,3-Dichlorobiphenyl	525	51	34-136	0-40	5.0
2,4,5-Trichlorobiphenyl	525	51	10-150	0-50	5.0
2,2',4,4'-Tetrach(orobiphenyl (MS)	525	51	10-150	0-50	5.0
2,2',3',4,6-Pentachtorobiphenyl	525	51	10-150	0-50	5.0
2,2',4,4',5,6'-Hexachtorobiphenyt	525	51	10-150	0-50	5.0
2,2',3,3',4,4',6-Heptachlorobiphenyl	525	51	10-150	0-50	5.0
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	525	51	10-150	0-50	5.0
Surrogate - Perylene-d12	525	51	40-150	AK	HA
Surrogate - Pyrene-d10	525	51	40-150	NA NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PGL** (Ug/L)
Aldicarb	531.1	33/51	50-140	0-20	0.50
Aldicarb sulfone	531.1	33/51	55-130	0-20	0.50
Aldicarb sulfoxide	531.1	33/51	40-160	0-40	0.50
Carbaryl	531.1	33/51	55-130	0-30	1.0
Carbofuran	531.1	33/51	50-130	0-20	1.0
Ethylene thiourea	531.1***	51	40-140	0-50	10
3-Hydroxycarbofuran	531,1	33/51	60-140	0-20	1.0
Methiocarb	531.1	51	53-121	0-40	5.0
Methomyl	531.1	33/51	50-150	0-20	1.0
Oxamyl	531.1	33/51	40-160	0-30	1.0
Propoxur (Baygon)	531.1	33/51	50-125	0-40	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Glyphosate	547	51	40-160	0-50	50
Endothal	548	51	20-180	0-50	25
Diquat	549/HRS	51/56	10-150	0-50	1.0
Paraquat	549/HRS	51/56	10-150	0-50	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Senzyl chloride	8010(5030)	2	50-150	0-30	1.0
Bromobenzene	8010(5030)	2	70-130	0-30	10
Bromodichloromethane	601/8010(5030)	1/2	42-172	0-30	1.0
Bromoform	601/8010(5030)	1/2	13-159	0-30	5.0
Bromomethane	601/8010(5030)	1/2	10-144	0-30	1.0
Carbon tetrachloride	601/8010(5030)	1/2	43-143	0-30	1.0
Chlorobenzene (MS)	601/8010(5030)	1/2	25-134	0-29	1.0
Chloroethane	601/8010(5030)	1/2	46-137	0-30	1.0
Chloreform	601/8010(5030)	1/2	49-133	0-30	1.0
1-Chioronexane	8010(5030)	2	50-150	0-30	1.0
2-Chloroethylvinyl ether	601/8010(5030)	1/2	14-186	0-80	10
Chloromethane	601/8010(5030)	1/2	10-193	0-30	1.0
Chiorotoluenes	8010(5030)	2	70-130	0-30	10
Dibromochloromethane	601/8010(5030)	1/2	24-191	0-30	1.0
Dibromomethane	8010(5030)	2	70-130	0-30	5.0
1,2-Dichlorobenzene	601/8010(5030)	1/2	10-208	0-30	1.0
1,3-Dichlorobenzene	601/8010(5030)	1/2	10-187	0-30	1.0
1,4-Dichlorobenzene	601/8010(5030)	1/2	42-143	0-30	1,0
Dichlorodifluoromethane	601/8010(5030)	1/2	70-130	0-30	1.0
1,1-Dichloroethane	601/8010(5030)	1/2	47-132	0-30	1.0
1,2-Dichloroethane	601/8010(5030)	1/2	51-147	0-30	1.0
1,1-Dichloroethene (MS)	601/8010(5030)	1/2	55-133	0-29	1.0
cis/trans-1,2-Dichloroethene	601/8010(5030)	1/2	38-155	0-30	1.0
Dichloromethane (methylene chloride)	601/8010(5030)	1/2	25-162	0-30	1,0
1,2-Dichloropropane	601/8010(5030)	1/2	44-156	0-30	1.0
cis/trans-1,3-Dichloropropylene	601/8010(5030)	1/2	22-178	0-30	1.0
1,1,2,2-Tetrachloroethane	601/8010(5030)	1/2	10-184	0-30	1.0
1,1,1,2-Tetrachloroethane	8010(5030)	2	70-130	0-30	1.0
Tetrachloroethylene	601/8010(5030)	1/2	26-162	0-30	1.0
1,1,1-Trichloroethane	601/8010(5030)	1/2	41-138	0-30	1.0
1,1,2-Trichloroethane	601/8010(5030)	1/2	39-136	0-30	1.0
Trichloroethene (MS)	601/8010(5030)	1/2	51-142	0-24	1.0
Trichlorofluoromethane	601/8010(5030)	1/2	21-156	0-30	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
1,2,3-Trichloropropane	8010(5030)	2	50-150	0-30	1_0
Vinyl chloride	601/8010(5030)	1/2	28-163	0-30	1.0
1,2-Dibromoethane (ED8)	8010***(5030)'	2	75-125	0-30	1.0
Surrogate - Bromochloromethane	601/8010(5030)	1/2	46-118	NA NA	NA

^{&#}x27; EDB determined on Hall detector with PQL of 1.0 ug/L at client's request.

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	жЕТНОD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Acetone	8015***(5030)	2	40-130	0-30	25
2-Butanone (MEK)	8015(5030)	2	60-130	0-40	25
Diethyl ether	8015(5030)	2	10-130	0-50	25
Ethanol	8015(5030)	2	20-140	0-45	1000
Ethyl methacrylate	8015***(5030)	2	42-125	0-40	10
Isobutanol	8015***(5030)	2	50-125	0-40	1000
Isopropanol	8015***(5030)	2	30-140	0-40	1000
Methacrylonitrile	8015***(5030)	2	10-140	0-60	100
Methanol	8015***(5030)	2	50-150	0-40	1000
Methyl methacrylate	8015***(5030)	2	45-132	0-42	10
4-Methyl-2-pentanone (MIBK)	8015(5030)	2	65-125	0-40	25
Methyl t-butyl ether (MTBE)	8015***(5030)	2	50-150	0-30	10
Propionitrile	8015***(5030)	2	10-130	0-50	100
Gasotine	8015 (modified)	12	40-140	0-40	50
Mineral spirits	8015 (modified)	12	40-140	0-40	50
Methanol (MS)	8015 (modified/DAI*)	2	50-150	0-50	1000
Ethanol	8015 (modified/DAI*)	2	50-150	0-50	1000
n-Procanol	8015 (modified/DAI*)	2	50-150	0-50	1000
(MS)	8015 (modified/DAI*)	2	50-150	0-50	1000
n-Butanol	8015 (modified/DAI*)	2	50-150	0-50	1000
Isobutanol	8015 (modified/DAI*)	2	50-150	0-50	1000
Ethylene glycol (MS)	8015 (modified/DAI*)	2	50-150	0-50	10000
Propylene glycol	8015 (modified/DAI*)	2	50-150	0-50	10000
Diethylene glycol	8015 (modified/DAI*)	2	50-150	0-50	10000
Triethylene glycol	8015 (modified/DAI*)	2	50-150	0-50	10000
Tetraethylene glycol	8015 (modified/DAI*)	2	50-150	0-50	25000

^{*} DAI = Direct Aqueous Injection

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Benzene (MS)	602/8020(5030)	1/2	61-131	0-25	1.0
Chlorobenzene (MS)	602/8020(5030)	1/2	54-136	0-24	1.0
1,2-Dichtorobenzene	602/8020(5030)	1/2	37-154	0-30	1.0
1,3-Dichtorobenzene	602/8020(5030)	1/2	50-141	0-30	1.0
1,4-Dichlorobenzene	602/8020(5030)	1/2	42-143	0-30	1.0
Ethylbenzene	602/8020(5030)	1/2	32-160	0-30	1.0
Methyl t-butyl ether	602/8020***(5030)	1/2	50-150	0-30	10
Toluene (MS)	602/8020(5030)	1/2	64-144	0-29	1.0
Xylenes	602***/8020(5030)	2	50-150	0-30	1.0
Surrogate - a,a,a-Trifluorotoluene	602/8020(5030)	1/2	77-140	NA AK	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	POL** (ug/L)
Acrolein	603/8030(5030)	1/2	88-118	0-30	200_
Acrylonitrile	603/8030(5030)	1/2	71-135	0-30	100
Acetonitrile	8030***(5030)	2	20-115	0-30	1000

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION*	PQL**
2-Chlorophenol (MS)	604/8040(3520)	1/2	30-111	0-26	10
4-Chloro-3-methylphenol (MS)	604/8040(3520)	1/2	41-107	0-23	10
2,4-Dichlorophenol	604/8040(3520)	1/2	44-119	ზ-40	10
2,4-Dimethylphenot	604/8040(3520)	1/2	24-118	0-40	10
2,4-Dinitrophenol	604/8040(3520)	1/2	12-145	0-65	50
2-Methyl-4,6-dinitrophenol	604/8040(3520)	1/2	30-136	0-40	50
3-Methyl phenol (m-cresol)	***8040(3520)	2	10-150	0-50	10
2-Methyl phenol (o-cresol)	***8040(3520)	2	10-150	0-50	10
4-Methyl phenol (p-cresol)	***8040(3520)	2	10-150	0-50	10
Cresols	8040(3520)_	2	NA_	NA	10
2-Nitrophenol	604/8040(3520)	1/2	43-117	0-40	10
4-Nitrophenol (MS)	604/8040(3520)	1/2	10-140	0-40	50
Pentachlorophenol (MS)	604/8040(3520)	1/2	10-135	0-41	50
Phenol (MS)	604/8040(3520)	1/2	10-122	0-60	10
Trichlorophenols	8040(3520)	2	NA.	NA	10
2,3,4,5-Tetrachlorophenol	***8040(3520)	2	50-150	0-40	20_
2,3,4,6-Tetrachlorophenol	***8040(3520)	2	50-150	0-40	20
Tetrachlorophenois	8040(3520)	22	NA	NA	20
2,4,5-Trichtorophenol	***8040(3520)	2	53-119	0-40	10
2,4,6-Trichlorophenol	604/8040(3520)	1/2	53-119	0-40	10
Surrogate - 2,4,6-Tribromophenol	604/8040(3520)	1/2	32-160	NA .	NA.

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Dichlorophen	604.1	18	22-125	0-30	10
Hexachlorophene	604.1	18	73-125	0-30	10

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Bis(2-ethylhexyl) phthalate (MS)	606/8060(3520)	1/2	10-162	0-82	10
Butyl benzyl phthalate (MS)	606/8060(3520)	1/2	10-137	0-73	10
Diethyl phthalate (MS)	606/8060(3520)	1/2	10-142	0-47	10
Dimethyl phthalate (MS)	606/8060(3520)	1/2	10-158	0-63	10
Di-n-butyl phthalate (MS)	606/8060(3520)	1/2	18-137	0-46	10
Di-n-octyl phthalate (MS)	606/8060(3520)	1/2	12-145	0-52	10
Surrogate - 2-Fluorobiphenyl	606/8060(3520)	1/2	27-123	NA.	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Aldrin (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	42-116 40-120	0-25 0-22	0.050 0.050
Benfluratin	608/8080(3520)***	1/2	40-140	0-40	0.010
alpha BHC	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	37-134	0-40	0.050 0.050
beta BHC	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	17-147	0-40	0.050 0.050
delta BHC	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	19-140	0-40	0.050 0.050
gamma BHC (Lindane) (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	52-136 56-123	0-18 0-15	0.050 0.050
Carbophenothion	608/8080***(3520)	1/2	50-110	0-40	1.0
alpha Chlordane	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	45-140	0-40	0.050 0.50 0.050
gamma Chlordane	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	45-140	0-40	0.050 0.50 0.050
technical Chlordane	608/8080(3520)/617	1/2/26	45-119	0-40	0.50
Chlorobenzilate	8081***V(3520)	2	50-150	0-40	0.50
Chlorothalonil	608/8080***(3520)	1/2	55-125	0-30	0.20
4,41-000	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	31-141	0-50	0.10 0.10
4,4'-DDE	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	30-145	0-50	0.10 0.10
4,4'-DOT (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	67-137 38-127	0-28 0-27	0.10 0.10
Dicofol (Kelthane)	8081***V(3520)	1/2	55-115	0-40	0.050
Dieldrin (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	51-143 52-126	0-46 0-18	0.10 0.10
Endosulfan I	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	45-153	0-40	0.050 0.050
Endosulfan II	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	10-202	0-65	0.10 0.10
Endosulfan sulfate	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	26-144	0-50	0.10 0.10
Endrin (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	57-142 56-121	0-23 0-21	0.10 0.1 0
Endrin aldehyde	608/8080(3520)/617 CLP - 3/90	1/2/26 62	10-150	0-50	0.10 0.10
Endrin ketone	CLP - 2/88; 3/90	6/62	NA	AK	0.10

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Heptachlor (MS)	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	42-129 40-131	0-2 2 0-20	0.050 0.050
Heptachlor epoxide	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	37-142	0-40	0.050 0.050
Isodrin	8081***V(3520)	2	55-110	0-40	0.050
Kepone	8081***V(3520)	2	10-150	0-50	0.10
Methoxychlor	8080(3520)/617 CLP - 2/88; 3/90	2/26 6/62	50-140	0-40	0.50 0.50
Mirex	8081***V(3530)	2	52-112	0-37	0.50
Toxaphene	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	41-140	0-50	5.0 1.0 5.0
Trifluralin	608/8080***(3520)	1/2	54-124	0-40	0.010
PCB 1016	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	69-107	0-21	1.0 0.50 1.0
PC8 1221	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	15 - 178	0-20	2.0 0.50 2.0
PCB 1232	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	10-215	0-20	1.0 0.50 1.0
PCB 1242	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	39-150	0-20	1_0 0.50 1.0
PCB 1248	608/8080(3520)/617 CLP - 2/88 CLP - 3/90	1/2/26 6 62	38-158	0-20	1.0 0.50 1.0
PCB 1254	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	66-122	0-23	1.0
PCB 1260	608/8080(3520)/617 CLP - 2/88; 3/90	1/2/26 6/62	58-122	0-20	1.0 1.0
Surrogate - Dibutylchlorendate (DBC)	608/8080(3520)/617 CLP - 2/88	1/2/26	28-151 24-154	AK AK	NA NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	608/8080(3520)/617 CLP - 3/90	1/2/26 62	22-126 60-150	NA NA	NA NA
Surrogate - Decachlorobiphenyl (DCB)	608/8080(3520)/617 CLP - 3/90	1/2/26	25-126 '60-150	NA NA	HA HA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMÉTER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Chloroneb	608.1	10	49-125	0-30	0.4
Chloropropylate	608.1	10	51-125	0-30	0.5
Chlorobenzilate (MS)	608.1	10	53-125	0-30	0.5
Etridiazole	608.1	10	60-125	0-30	0.01
PCNB	608.1	10	60-125	0-30	0.6
Propachior	608.1	10	51-125	0-30	0.5
Chiorothalonil	608.2	57	55-125	0-30	0.20
DCPA (Dacthal)	608.2	57	50-150	0-40	0.50
Dichloran	608.2	57	56-110	0-40	5.0
Methoxychlor	608.2	57	50-140	0-40	0.50
Permethrin	608.2	57	50-130	0-40	1.0
Surrogate - Dibutylchlorendate (DSC)	608.1/608.2	10/57	28-151	NA	NA .
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	608.1/608.2	10/57	22-126	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
2,4-Dinitrotoluene (MS)	609/8090(3520) (FID) 609/8090(3520) (ECD)	1,2	10-125 10-125	0-40 0-40	10 0.3
2,6-Dinitrotoluene (MS)	609/8090(3520) (FID) 609/8090(3520) (ECD)	1,2	10-126 10-126	0-40 0-40	10 0.3
Isophorone (MS)	609/8090(3520)	1,2	10-117	0-40	10
Nitrobenzene (MS)	609/8090(3520)	1,2	10-118	0-40	10
Surrogate - 2-fluorobiphenyl	609/8090(3520) (FID)	1,2	27-123	NA NA	NA
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	609/8090(3520) (ECD)	1,2	22-126	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Acenaphthene (MS)	610/8100(3520)	1/2	38-111	0-21	10
Acenaphthylene	610/8100(3520)	1/2	38-110	0-23	10
Benzo(a)pyrene (MS)	610/8100(3520)	1/2	24-132	0-78	10
Benzo(b+k)fluoranthene	610/8100(3520)	1/2	28-129	0-65	10
Benzo(g,h,i)perylene	610/8100(3520)	1/2	14-147	0-87	10
Carbazole	8100***(3520)	2	16-140	0-40	10
Chrysene + Benzo(a)anthracene	610/8100(3520)	1/2	29-129	0-68	10
fluoranthene	610/8100(3520)	1/2	12-155	0-67	10
Fluorene (MS)	610/8100(3520)	1/2	39-115	0-23	10_
Indeno(1,2,3-cd) pyrene + Dibenzo(a,h)anthracene	610/8100(3520)	1/2	15-151	0-87	10
1-Methyl naphthalene	610/8100(3520)	1/2	20-140	0-50	10_
2-Methyl naphthalene	610/8100(3520)	1/2	20-140	0-50	10
Waphthalene (MS)	610/8100(3520)	1/2	34-103	0-25	10
Phenanthrene + Anthracene	610/8100(3520)	1/2	38-119	0-29	10
Pyrene (HS)	610/8100(3520)	1/2	36-124	0-37	10
Diesel	8100 (modified)	12	40-140	0-40	300
Surrogate - 2-Fluorobiphenyl	610/8100(3520)	1/2	27-123	NA.	NA_
Surrogate - Decafluorobiphenyl	8100 (modified) (3520)	12	20-150	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Acephate	614***/8141***(3520)	52/2	25-140	0-50	5.0
Azinphos methyl (MS)	614/622/8141(3520)	14/2	16-129	0-50	1.0
Bolstar (MS)	622/8141(3520)	14/2	58-156	0-40	1.0
Carbophenothion	8141***(3520)	2	20-150	0-40	1.0
Chlorpyrifos	614/622/8141(3520)	52/14/2	82-115	0-40	1.0
Chlorpyrifos methyl	622	14	20-130	0-40	1.0
Cournachos	622/8141(3520)	14/2	51-147	0-40	1.0
Demeton-o	614/622/8141(3520)	52/14/2	36-120	0-40	2.5
Demeton-s	614/622/8141(3520)	52/14/2	36-120	0-40	2.5
Diazinon (MS)	614/622/8141(3520)	52/14/2	36-124	0-40	1.0
Dichlofenthion	614/8141***V(3520)	52/2	62-104	0-40	1.0
Dichlorvos	622/8141(3520)	14/2	49-120	0-40	2.0
Dimethoate	8141(3520)	2	38-120	0-40	10
Dioxathion	614/8141***V/3520)	52/2	25-140	0-40	10
Disulfoton (MS)	614/622/8141(3520)	52/14/2	10-178	0-66	2.0
EPN	614.1/8141(3520)	58/2	48-124	0-40	1.0
Ethion	614/614.1/8141(3520)	52/58/2	40-138	0-40	0.50
Ethoprop	622/8141(3520)	52/14/2	58-113	0-40	0.50
Famphur	8141***V(3520)	2	10-129	0-60	2.0
Fenamiphos	614***	52	40-160	0-40	0.50
Fensulfothion	622/8141(3520)	14/2	43-145	0-40	5.0
Fenthion	622/8141(3520)	14/2	10-128	0-60	1.0
Isofenphos	614***	52	40-160	0-40	0.50
Malathion	614/8141(3520)	52	60-140	0-40	1.0
Merphos	622/8141(3520)	14/2	50-130	0-40	1.0
Methamidophos	614***	52	40-160	0-40	2.0
Metolachlor	614/8141***(3520)	52/2	53-133	0-40	1.0
Mevinphos	622/8141(3520)	52/14/2	34-125	0-40	2.0
Monocrotophos	8141(3520)	52/2	25-140	0-50	10
Waled	622/8141(3520)	14/2	54-102	0-40	5.0
Parathion, ethyl (MS)	614/8141(3520)	52/2	18-171	0-28	1.0
Parathion, methyl (MS)	614/622/8141(3520)	52/14/2	40-104	0-40	0.50
Phorate (MS)	622/8141(3520)	14/2	36-125	0-40	1.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Ronnet	622/8141(3520)	14/2	45-135	0-35	1.0
Stirophos (Tetrachlorvinphos)	622/8141(3520)	14/2	48-125	0-40	1.0
Sulfoteop (MS)	8141(3520)	2	10-241	0-40	0.50
Terbufos	614.1	58	40-160	0-40	0.50
Thionazin	8141***V(3520)	2	25-160	0-60	1.0
Tokuthion (Prothiofos)	622/8141(3520)	14/2	44-125	0-40	1.0
Trichloronate	622/8141(3520)	14/2	49-161	0-40	1.0
Surrogate - Ronnel	8141/(3520)	2	45-135	NA	NA
Surrogate - Tokuthion	622/8141	14/2	44-125	NA	AK
Surrogate • Triphenylphosphate	614/622/8141	14/20/2	40-125	NA .	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL**
2,4-D (MS)	615/8150	53/2	25-129	0-60	0.50
2,4-DB	615/8150	53/2	40-140	0-40	0.50
2,4,5-T (MS)	615/8150	53/2	25-145	0-62	0.50
2,4,5-TP (Silvex) (MS)	615/8150	53/2	10-151	0-81	0.50
Dalapon	615/8150	53/2	10-160	0-80	10
Dicamba	615/8150	53/2	10-150	0-80	5.0
Dichlorprop	615/8150	53/2	10-150	0-80	0.50
Dinoseb	615/8150	53/2	10-150	0-80	0.50
МСРА	615/8150	53/2	10-150	0-80	10_
МСРР	615/8150	53/2	10-150	0-80	10
Pentachlorophenol	615/8150***	53/2	10-150	0-80	0.10
Pictoram	615/8150***V	53/2	10-150	0-40	0.050
Surrogate - 2,4-Dichlorophenylacetic acid (DCAA)	615/8150	53/2	10-135	NA	NA
Surrogate - 2,4-Dichlorophenoxy butyric acid (2,4-DB)	615/8150	53/2	40-140	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Benfluratin	617***	26	40-140	0-40	0.010
Captan (MS)	617	26	55-125	0-40	0.10
Carbophenothion	617	26	50-110	0-40	1.0
Chlorothalonil	617***	26	55-125	0-30	0.20
Dichloran	617	26	56-110	0-40	5.0
Dicafol	617	26	55-115	0-40	0.10
Isodrin (MS)	617	26	55-110	0-40	0.050
Mirex	617	26	54-104	0-40	0.50
PCNB	617	26	54-100	0-40	0.01
Pendimethalin	617***	26	52-128	0-40	2.0
Permethrin	617***	26	50-130	0-40	1.0
Perthane	617	26	55-115	0-40	5.0
Strobane	617	26	48-127	0-40	2.0
Irifluralin	617	26	54-124	0-40	0.01
Chloropicrin	618	27	62-134	0-40	1.0
Ethylene dibromide	618	27	48-90	0-40	0.50
Surrogate - 2,4,5,6-Tetrachloro-m-xylene (TCMX)	617/618	26/27	22-126	NA	AK

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Alachlor	619***	7	45-140	0-30	2.0
Ametryn	619	7	60-120	0-40	2.0
Atraton	619	7	50-115	0-40	5.0
Atrazine (MS)	619	7	40-125	0-30	2.0
Bromacil	619***	7	55-127	0-30	2.0
Hexazinone	619***	7	50-130	0-30	2.0
Metalaxyl	619***	7	50-130	0-40	1.0
Metribuzin	619***	7	61-141	0-30	2.0
Norflurazon	619***	7	54 - 134	0-30	2.0
Prometon	619	7	55-100	0-40	2.0
Prometryn	619	7_	55-120	0-40	2.0
Propazine (MS)	619	7	33-100	0-40	2.0
Secburieton	619	7	30-130	0-45	5.0
Simetryn	619	7	50-200	0-40	2.0
Simazine	619	7	25-174	0-50	2.0
Terbuthylazine	619	7	60-130	0-40	2.0
Terbutryn	619	7	53-113	0-40	2.0
Triadimefon	619***	7	61-125	0-30	2.0
Diphenylamine	620	23	56-125	0-30	2.0
Surrogate - Triphenylphosphate	619/620	7/23	40-125	NA NA	NA.

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	POL** (Ug/L)
Aspon	622.1	8	62-104	0-40	1.0
Dichlofenthion	622.1	8	62-104	0-40	1.0
Famphur	622.1	8	10-129	0-40	2.5
Fenitrothion	622.1	8	61-103	0-40	2.0
Fonophos	622.1	8	53-133	0-40	1.0
Phosmet	622.1	88	50-150	0-40	1.0
Thionazin	622.1	8	25-160	0-40	1.0
Surrogate - Triphenylphosphate	622.1	8	40-125	NA	ЖA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPO)	PQL** (ug/L)
Acetone	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	10-161	0-40	50 10
Acetonitrile	82408(5030)	2	52-170	0-40	1000
Acrolein	8240(5030)/8260A(5030)	2	60-132	0-40	100
Acrylonitrile	8240(5030)/8260A(5030)	2	77-108	0-40	100
Benzene (MS)	624/8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	1/2 6 62	73-144 76-127 76-127	0-22 0-11 0-11	5.0 5.0 10
Benzyl Chloride	8240B(5030)	2	10-130	0-70	100
Bromobenzene	8260A(5030)	2	50-150	0-40	10
Bromodichloromethane	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	35-15 5	0-40	5.0
Bromoform	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	45-169	0-40	5.0
Bromomethane	624/8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	1/2/6/62	10-242	0-65	10
2-Butanone (MEK)	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	79-208	0-40	50 10
n-Butylbenzene	. 8260A(5030)	2	50-150	0-40	5.0
sec-Butylbenzene	8260A(5030)	2	50-150	0-40	5.0
tert-Butylbenzene	8260A(5030)	2	50-150	0-40	5.0
Carbon disulfide	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6	37-138	0-40	5.0
Carbon tetrachloride	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	70-140	0-40	5.0
Chlorobenzene (MS)	624/8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	1/2 6 62	68-136 75-130 75-130	0-17 0-13 0-13	5.0 5.0 10
2-Chloro-1,3-butadiene (Chloroprene)	82408(5030)	2	21-163	0-50	5
Chloroethane	624/8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	1/2/6/62	64-119	0-65	10
2-Chloroethyl vinyl ether	624/8240(5030)/8260A(5030)	1/2	10-305	0-65	50
Chloraform	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	51-138	0-40	5.0
Chloromethane	624/8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	1/2/6/62	10-273	0-65	10
3-Chloropropene (Allyl chloride)	8240B(5030)	2	81-112	0-40	5
2-Chlorotoluene	624/8240***(5030)/8260A(5030)	1/2	58-125	0-40	5.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
4-Chlaratoluene	8260A (5030)	2	50-150	0-40	5.0
Dibromochloromethane	624/8240/CLP-2/88/8260A(5030) CLP-3/90	1/2/6 62	53-149	0-40	5.0 10
1,2-Dibromo-3-chloropropane (DBCP)	8240B(5030)/8260A(5030)	2	37-127	0-40	10
1,2-Dibromoethane	8240B(5030)/8260A(5030)	2	70-112	0-40	5.0
Dibromomethane	8240B(5030)/8260A(5030)	2	78-110	0-40	5.0
1,2-Dichtorobenzene	624/8240(5030)/8260A(5030)	1/2	69-112	0-40	5.0
1,3-Dichtorobenzene	624/8240(5030)/8260A(5030)	1/2	32-180	0-40	5.0
1,4-Dichlorobenzene	624/8240(5030)/8260A(5030)	1/2	39-158	0-40	5.0
trans-1,4-Dichloro-2-butene	8240B(5030)	2	11-129	0-40	10
Dichlorodifluoromethane	8240B(5030)/8260A(5030)	2	72-146	0-40	5.0
1,1-Dichloroethane	624/8240(5030)/CLP-2/88/	1/2/6	59-155	0-40	5.0
	8260A(5030) CLP-3/90	62			10
1,2-Dichloroethane	624/8240(5030)/CLP-2/88/	1/2/6	49-155	0-40	5.0
	8260A(5030) CLP-3/90	62			10
cis/trans-1,2-Dichloroethene	624/8240(5030)/CLP-2/88/	1/2/6	54-156	0-40	5.0
	8260A(5030) CLP-3/90	62			10
1,1-Dichloroethene (MS)	624/8240(5030)/CLP-2/88/	1/2/6	60-136	0-19	5.0
·	8260A(5030) CLP-3/90	62			10
1,2-Dichloropropane	624/8240(5030)/CLP-2/88/	1/2/6	10-210	0-65	5.0
	8260A(5030) CLP-3/90	62			10
1,3-Dichloropropane	8260A(5030)	2	50-150	0-40	5.0
2,2-Dichloropropane	8260A(5030)	2	50-150	0-40	5.0
1,1-Dichloropropene	8260A(5030)	2	50-150	0-40	5.0
cis-1,3-Dichloropropene	624/8240(5030)/CLP-2/88/	1/2/6	10-227	0-65	5.0
	8260A(5030) CLP-3/90	62			10
trans-1,3-Dichloropropene	624/8240(5030)/CLP-2/88/	1/2/6	17-183	0-65	5.0
cross 1,2 brancoropropene	8260A(5030) CLP-3/90	62			10
Ethanol	8240(5030)	2	40-160	0-40	1000
Ethylbenzene	624/8240(5030)/CLP-2/88/	1/2/6	37-162	0-40	5.0
, to chiech	8260A(5030)	1			10
Ethyl methacrylate	CLP-3/90 8240(5030)	62	37-139	0-40	5.0
Hexachlorobutadiene	8260A(5030)	2	50-150	0-40	5.0
2-Hexanone	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	10-164	0-40	50 10

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Iodomethane	82408(5030)/8260A(5030)	2	37-137	0-40	5.0
Isobutyl alcohol	82408(5030)	2	51-179	0-40	1000
Isopropylbenzene	8260A(5030)	2	50-150	0-40	5.0
p-Isopropyltoluene	8260A(5030)	2	50-150	0-40	5.0
Methacrylonitrile	82408(5030)	2	76-111	0-40	100
Methylene chloride	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6 62	10-221	0-65	5.0
Methylmethacrylate	82408(5030)	2	50-130	0-40	5.0
4-Methyl-2-pentanone (MIBK)	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	68-111	0-40	50 10
Methyl t-butyl ether (MTSE)	8240***(5030)	2	50-150	0-40	10
Naphthalene	8260A(5030)	2	50-150	0-40	5.0
Pentachloroethane	82408(5030)	2	10-276	0-65	25
Propionitrile (ethyloyanide)	82408(5030)	2	63-112	0-40	100
n-Propylbenzene	8260A(5030)	2	50-150	0-40	5.0
Styrene	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	60-109	0-40	5.0
	CLP-3/90	62.			10
1,1,1,2-Tetrachloroethane	8240B(5030)/8260A(5030)	2	34-138	0-40	5.0
1,1,2,2-Tetrachloroethane	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	46-157	0-40	5.0
Tetrachloroethene	624/8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	1/2/6	44-148	0-40	5.0
Totuene (MS)	624/8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	1/2 6 62	68-138 76-125 76-125	0-17 0-13 0-13	5.0 5.0 10
1,2,3-Trichlorobenzene	8260A(5030)	2	50-150	0-40	5.0
1,2,4-Trichlorobenzene	8260A(5030)	2	50-150	0-40	5.0
1,1,1-Trichloroethane	624/8240(5030)/CLP-2/88/ 8260A(5030)	1/2/6	52-162	0-40	5.0
1,1,2-Trichloroethane	624/8240(5030)/CLP-2/88/	1/2/6	52-150	0-40	10 5.0
	8260A(5030) CLP-3/90	62			10
Trichloroethene (MS)	624/8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	1/2 6 62	66-136 71-120 71-120	0-20 0-14 0-14	5.0 5.0 10
Trichlorofluoromethane	8240(5030)/8260A(5030)	2	17-181	0-65	5.0
1,2,3-Trichloropropane	8240(5030)/8260A(5030)	2	44-103	0-40	5.0
Trichlorotrifluoroethane	8240***(5030)	2	82-130	0-23	5.0
1,2,4-Trimethylbenzene	8260A(5030)	2	50-150	0-40	5.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	HETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
1,3,5-Trimethylbenzene	8260A(5030)	2	50-150	0-40	5.0
Vinyl acetate	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	49-147	0-40	10
Vinyl chloride	624/8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	1/2/6/62	10-251	0-65	10
Xylenes	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	66-114	0-40	5.0
	CLP-3/90	62			10
Surrogate - Toluene-d8	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	77-120 88-110	NA NA	NA AK
Surrogate - p-Bromofluorobenzene	624/8240(5030)/8260A(5030) CLP-2/88; 3/90	1/2 6/62	80-125 86-115	NA NA	HA NA
Surrogate - Dibromofluoromethane	8260A(\$030)	2	86-118	NA	NA
Surrogate - 1,2-Dichloroethane-d4	624/8240(5030) CLP-2/88; 3/90	1/2 6/62	80-125 76-114	NA NA	NA NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Acenaphthene (MS)	625/82708(3520) CLP-2/88; 3/90	1/2 6/62	65-116 46-118	0-20 0-31	10 10
Acenaphthylene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	54-130	0-24	10
Acetophenone	8270(3520)	2	10-150	0-50	10
2-Acetylaminofluorene	8270(3520)	2	25-150	0-50	10
Aldrin	625/8270(3520)	1/2	10-166	0-40	10
4-Aminobiphenyl	8270(3520)	2	10-150	0-50	10
Aniline	8270(3520)	2	10-150	0-50	50
Anthracene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	59-125	0-19	10
Aramite	8270(3520)	2	40-150	0-50	10
Benzidine	625/8270(3520)	1/2	10-200	0-100	80
Benzoic acid	8270(3520)/CLP-2/88	2/6	10-150	0-50	50
Benzo(a)anthracene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	54-125	0-20	10
8enzo(b)fluoranthene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	45-135	0-21	10
Benzo(k)fluoranthene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	40-160	0-33	10
Benzo(g,h,i)perylene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-219	0-50	10
Benzo(a)pyrene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	54-137	0-21	10
Benzyl alcohol	8270(3520)/CLP-2/88	2/6	10-150	0-50	10
Benzyl chloride	82708***(3520)	2	10-150	0-50	10
alpha-BHC	625/8270(3520)	1/2	10-150	0-50	10
beta-BHC	625/8270(3520)	1/2	24-149	0-40	10
delta-BHC	625/8270(3520)	1/2	10-110	0-40	10
gamma - BHC	625/8270(3520)	1/2	10-150	0-50	10
Bis(2-chloroethoxy) methane	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	33-184	0-50	10
Bis(2-chloroethyl) ether	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	12-158	0-50	10
Bis(2-chloroisopropyl) ether	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	36-166	0-50	10
Bis(2-ethylhexyl) phthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-158	0-40	10
4-Bromophenyl phenyl ether	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	53-127	0-40	10
Butyl benzyl phthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-152	0-40	10
Carbazole	8270(3520)***/CLP-3/90	2/62	10-150	0-50	10
technical Chlordane	625/8270(3520)	1/2	10-150	0-50	50
p-Chloroaniline	8270(3520) CLP-2/88; 3/90	2 6/62	10-150	0-50	20
4-Chloro-3-methyl-phenol (MS)	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	53-104 23-97	0-17 0-42	10

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
1-Chloronaphthalene	8270(3520)	2	10-150	0-50	10
2-Chloronaphthalene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	60-118	0-40	10
2-Chlorophenol (MS)	625/8270(3520) CLP-2/88; 3/90	1/ 6/62	54-99 27-123	0-18 0-40	10 10
4-Chlorophenylphenyl ether	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	25 - 1 <u>5</u> 8	0-33	10
Chrysene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	45-131	0-25	10
3-Methyl phenol (m-Cresol)	8270(3520)	2	10-150	0-50	10
2-Methyl phenol (o-Cresol)	8270(3520)/CLP-2/88; 3/90	2/6/62	10-150	0-50	10
4-Methyl phenol (p-Cresol)	8270(3520)/CLP-2/88; 3/90	2/6/62	10-150	0-50	10
4,41-000	625/8270(3520)	1/2	10-145	0-40	10
4,41-00E	625/8270(3520)	1/2	10-136	0-40	10
4,4'-DOT	625/8270(3520)	1/2	10-203	0-62	10
Diallate	8270(3520)	2	10-150	0-50	10
Dibenz(a,h)anthracene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	47-135	0-24	10
Dibenzofuran	8270(3520)/CLP-2/88; 3/90	2/6/62	10-150	0-50	10
Di-n-butyl phthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-118	0-50	10
1,2-Dichlorobenzene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	32-129	0-40	10
1,3-Dichlorobenzene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-172	0-42	10
1,4-Dichlorobenzene (MS)	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	46-110 36-97	0-18 0-28	10 10
3,3'-Dichtorobenzidine	625/8270(3520)/CLP-2/88 CLP 3/90	1/2/6 62	10-262	0-100	20 10
2,4-Dichlorophenol	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	39-135	0-40	10
2,6-Dichlorophenol	8270(3520)	2	10-150	0-50	10
Dieldrin	625/8270(3520)	1/2	29-136	0-40	10
Diethyl phthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-114	0-40	10
p-(Dimethylamino)azobenzene	8270(3520)	2	10-150	0-50	10
7,12- Dimethylbenz(a)anthracene	8270(3520)	2	10-150	0-50	10
3,3'-Dimethylbenzidine	8270(3520)	2	10-200	0-100	200
a,a-Dimethylphenethylamine	8270(3520)	2	10-200	0-50	2000
2,4-Dimethylphenol	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	44-112	0-25	10
Dimethylphthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-112	0-40	10
m-Dinitrobenzene	82708(3520)	2	10-150	0-50	10
4,6-Dinitro-2-methylphenol	625/8270(3520)/CLP-2/88 CLP 3/90	1/2/6	10-181	0-93	50 25

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
2,4-Dinitrophenol	625/8270(3520)/CLP-2/88 CLP 3/90	1/2/6 62	10-143	0-48	50 25
2,4-Dinitrotoluene (MS)	625/8270(3520) CLP-2/88; 3/90	1/2	39-133 24-96	0-25 0-38	10 10
2,6-Dinitrotoluene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	50-158	0-40	10
Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	8270(3520)	2	10-150	0-50	10
Di-n-octyl phthalate	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-146	0-50	10
1,4-Dioxane	8270***V(3520)	2	10-150	0-50	10
Diphenylamine/ N-nitrosodiphenylamine	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-150	0-50	10
1,2-Diphenyl hydrazine	8270(3520)	2	10-150	0-50	10
Endosulfan I	625/8270(3520)	1/2	10-150	0-50	20
Endosulfan II	625/8270(3520)	1/2	10-150	0-50	20
Endosulfan sulfate	625/8270(3520)	1/2	10-107	0-50	20
Endrin	625/8270(3520)	1/2	10-150	0-50	20
Endrin aldehyde	625/8270(3520)	1/2	10-209	0-50	50
Endrin ketone	8270/(3520)	2	10-150	0-50	50
Ethyl carbamate	8270***V(3520)	2	52-100	0-24	10
Ethyl methane sulfonate	8270(3520)	2	10-150	0-50	10
fluoranthene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	58-124	0-19	10
fluorene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	59-121	0-40	10
Heptachlor	625/8270(3520)	1/2	10-192	0-40	20
Meptachlor epoxide	625/8270(3520)	1/2	26-155	0-55	20_
Hexachlorobenzene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	10-152	0-40	10
Hexachlorobutadiene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	24-116	0-40	10
Hexachlorocyclopentadiene	8270(3520)/CLP-2/88; 3/90	2/6/62	10-150	0-50	10
Hexachloroethane	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	40-113	0-40	10
Hexachiorophene	8270(3520)	2	10-200	0-80	5000
Kexachloropropene	8270(3520)	2	10-150	0-50	10
Indeno(1,2,3-cd)pyrene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	33-147	0-36	10
Isophorone	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	21-196	0-60	10
Isosafrole	8270(3520)	2	10-150	0-50	10
Methapyrilene	8270(3520)	2	10-150	0-50	2000
3-Methylcholanthrene	8270(3520)	2	10-150	0-50	10

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	POL** (Ug/L)
<u>Hethylmethanesulfonate</u>	8270(3520)	2	10-150	0-50	10
2-Methylnaphthalene	8270(3520)/CLP-2/88; 3/90	2/6/62	10-150	0-50	10
1-Methylnaphthalene	8270B(3520)	2	10-150	0-50	10
Naphthalene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	46-111	0-19	10
1,4-Napthoquinone	8270(3520)	2	10-150	0-50	10
1-Napthylamine	8270(3520)	2	10-150	0-50	10
2-Napthylamine	8270(3520)	2	10-150	0-50	10
Nicotine	8270(3520)	2	10-150	0-50	100
2-Nitroaniline	8270(3520)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	50 25
3-Nitroaniline	8270(3520)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	50 25
4-Nitroaniline	8270(3520)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	50 25
Nitrobenzene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	35-180	0-40	10
2-Nitrophenol	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	29-182	0-40	10
4-Nitrophenol (MS)	625/8270(3520) CLP-2/88 CLP-3/90	1/2 6 62	10-112 10-80 10-80	0-108 0-50 0-50	50 50 25
4-Nitroquinoline-1-oxide	8270(3520)	2	10-150	0-50	100
N-Nitrosodi-n-butylamine	8270(3520)	2	10-150	0-50	10
N-Nitrosodiethylamine	8270(3520)	2	10-150	0-50	10
N-Nitrosodimethylamine	625/8270(3520)	1/2	10-150	0-50	10
N-Nitrosodi-n-propylamine (MS)	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	39-142 41-116	0-60 0-38	10 10
N-Nitrosomethylethylamine	8270(3520)	2	10-150	0-50	10
N-Mitrosomorpholine	8270(3520)	2	10-150	0-50	10
N-Nitrosopiperidine	8270(3520)	2	10-150	0-50	10
N-Nitrosopyrrolidine	8270(3520)	2	10-150	0-50	10
S-Nitro-o-toluidine	8270(3520)	2	10-150	0-50	10
PCB-1016	625/8270(3520)	1/2	10-150	0-50	500
PC8-1221	625/8270(3520)	1/2	10-150	0-50	500
PC8-1232	625/8270(3520)	1/2	10-150	0-50	500
PCB-1242	625/8270(3520)	1/2	10-150	0-50	500
PCB-1248	625/8270(3520)	1/2	10-150	0-50	500
PCB-1254	625/8270(3520)	1/2	10-150	0-50	500

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
PCB-1260	625/8270(3520)	1/2	10-150	0-50	500
Pentachlorobenzene	8270(3520)	2	10-150	0-50	10
Pentachloronitrobenzene	8270(3520)	2	10-150	0-50	10
Pentachlorophenol (MS)	625/8270(3520) CLP 2/88 CLP 3/90	1/2 6 62	15-139 9-103 9-103	0-39 0-50 0-50	50 50 25
Phenacetin	8270(3520)	2	10-150	0-50	10
Phenanthrene	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	54-119	0-20	10
Phenol (MS)	625/8270(3520) CLP 2/88 CLP 3/90	1/2 6 62	10-96 12-89 12-110	0-21 0-42 0-42	10 10 10
p-Phenylenediamine	8270(3520)	2	10-200	0-50	2000
2-Picoline	8270(3520)	2	10-150	0-50	200
Pronamide	8270(3520)	2	10-150	0-50	10
Pyrene (MS)	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	36-153 26-127	0-21 0-31	10 10
Pyridine	8270(3520)	2	10-150	0-50	200
Safrole	8270(3520)	2	10-150	0-50	10
Strychnine	8270(3520)	2	10-150	0-50	100
1,2,4,5-Tetrachlorobenzene	8270(3520)	2	10-150	0-50	10
Trichlorophenols	8270(3520)	2	NA	NA	10
2,3,4,5-Tetrachlorophenol	8270***(3520)	2	10-150	0-50	50
2,3,4,6-Tetrachlorophenol	8270(3520)	2	45-129	0-22	50
o-Toluidine	8270(3520)	2	10-150	0-50	10
Toxaphene	625/8270(3520)	1/2	10-200	0-80	2000
1,2,4-Trichlorobenzene (MS)	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	49-108 39-98	0-24 0-28	10 10
Tetrachlorophenols	8270(3520)	2	NA	NA	50
2,4,5-Trichlorophenol	8270(3520) CLP-2/88 CLP-3/90	2 6 62	45-113	0-21	10 50 25
2,4,6-Trichlorophenol	625/8270(3520)/CLP-2/88; 3/90	1/2/6/62	37-144	0-40	10
o,o,o-Triethyl- phosphorothicate	8270(3520)	2	10-150	0-50	10
1,3,5-Trinitrobenzene	8270(3520)	2	10-150	0-50	200
Surrogate - Nitrobenzene-d5	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	61-115 35-114	NA	NA NA

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PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Surrogate - 2-Fluorobiphenyl	625/8270(3520) CLP-2/88; 3/90	1/2 6/62	59-119 43-116	NA NA	NA NA
Surrogate - p-Terphenyl-d14	8270(3520) CLP-2/88; 3/90	2 6/62	46-136 33-141	NA NA	NA NA
Surrogate - Phenol-d5	625/8270(3520) CLP-2/88 CLP 3/90	1/2 6 62	10-106 10-94 10-110	NA NA NA	NA NA
Surrogate - 2-Fluorophenol	8270(3520) CLP-2/88 CLP 3/90	2 6 62	10-104 21-100 21-110	AA AK AK	NA NA NA
Surrogate - 2,4,6-Tribromophenol	8270(3520) CLP-2/88; 3/90	2 6/62	41-143 10-123	NA NA	NA NA
Surrogate - 2-Chlorophenol-d4	CLP-3/90	62	33-110	на	NA
Surrogate - 1,2-Dichiorobenzene-d4	CLP-3/90	62	16-110	NA	NA

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PARAMETER	METHOD (Prep)	REFERENCE	(% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
2,3,7,8-Tetrachlorodibenzo-p- dioxin (2,3,7,8-TCDD) (MS)	613/8280 8270 (Screen)	1/2	63-137	0-40	0.0050 10
Polychlorinated Dibenzo-p-dioxin	and Dibenzofuran clas	ses			
tetra-CDD (MS)	8280	2	63-137	0-40	0.0050
tetra-CDF (MS)	8280	2	60-142	0-40	0.0050
penta-CDD (MS)	8280	2	37-163	0-40	0.0050
penta-CDF (MS)	8280	2	52-148	0-40	0.0050
hexa-CDD (MS)	8280	2	42-158	0-40	0.0050
hexa-CDf (MS)	8280	_2	58-142	0-40	0.0050
hebta-CDD (MS)	8280	2	20-170	0-50	0.010
hepta-CDF (MS)	8280	2	20-170	0-50	0.010
octa-CDD (MS)	8280	2	20-170	0-50	0.010
octa-CDF (MS)	8280	2	20-170	0-50	0.010
Internal Standard - "C.,-2,3,7,8-TCDD	8280	2	40-120	NA	NA
Internal Standard - 13C12-OCDD	8280	2	40-120	NA	NA

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PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPO)	PQL**
Benfluralin	627	9	40-140	0-40	0.010
Ethalfluralin	627	9	40-140	0-40	2.0
Isopropatin	627	9	48-140	0-40	0.10
Profluration	627	9	55-140	0-40	0.20
Trifluralin (MS)	627	9	17-140	0-50	0.010
Surrogate - 2,4,5,6-Tetrachloro-m-xylene	627	9	22-126	NA NA	NA
Cyanazine	629	25	20-180	0-50	5.0
Benomyl	631	55	50-126	0-30	5.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL**
Amobam	630	63	70-130	0-20	
Ferbam	630	_63	70-130	0-20	•
Mancozeb	630	63	70-130	0-20	*
Maneb	630	63	70-130	0-20	•
Metham	630	63	70-130	0-20	•
Nabam	630	63	70-130	0-20	•
Polyram	630	63	70-130	0-20	
Zineb	630	63	70-130	0-20	
Ziram	630	63	70-130	0-20	20

^{*} All compounds reported as Ziram

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Aminocarb	632	13	60-125	0-30	1.0
Barban	632	13	55-125	0-30	1.0
Bromacil	632***V	13	52-125	0-30	2.0
Carbaryi (MS)	632	13	55-125	0-30	<u>5.</u> 0
Carbofuran	632	13	55-125	0-30	10
Chlorpropham	632	13	55-125	0-30	1.0
Diuron (MS)	632	13	55-125	0-30	1.0
Fenuron	632	13	60-125	0-30	5.0
Fluometuron	632	13	59-125	0-40	1.0
Linuron	632	13	55-125	0-30	1.0
Methiocarb	632	13	51-137	0-30	5.0
Methomyl	632	13	52-132	0-30	1.0
Monuron	632	13	56-132	0-30	1.0
Neburon	632	13	54-126	0-30	1.0
Oxamyt	632	13	57-125	0-30	10
Propham	632	13	50-125	0-30	1.0
Propoxur	632	13	56-125	0-30	1.0
Siduron	632	13	55-125	0-30	1.0
Swep	632	13	58-125	0-35	1.0
Surrogate - Propachlor	632	13	45-125	NA.	AK

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL**
Bromacil	633	41	52-125	0-30	2.0
DEET	633	41	55-125	0-30	5.0
Hexazinone	_633	41	52-125	0-30	0.50
Metribuzin	633	41	50-125	0-30	1.0
Terbacil	633	41	50-130	0-30	5.0
Triadimefon	633	41	48-125	0-30	1.0
Tricyclazole	633	41	53-125	0-30	5.0
Surrogate - Triphenylphosphate	633	41	40-125	АН	NA

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PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Butylate (MS)	634	15	38-145	0-76	2.0
Cycloate	634	15	46-159	0-47	2.0
EPTC	634_	15	46-154	0-55	2.0
Holinate (MS)	634	15	37-127	0-74	2.0
Pebulate	634	15	22-172	0-50	2.0
Vernolate	634	15	39-147	0-45	2.0
Surrogate - Tokuthion	634	15	44-125	NA :	NA
Surrogate - Triphenylphosphate	634	15	40-125	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)			
Rotenone	635	19	59-125	0-30	2.0			
Bensulide	636	16	22-140	0-50	2.0			
Oryzalin	638	21	50-130	0-30	1.0			
Bendiocarb	639	20	10-165	0-50	2.0			
Bentazon	643	59	50-150	0-40	5.0			
2,4,-0	644***	64	25-129	0-60	2.0			
2,4,-08	644***	64	48-126	0-40	1.0			
Dicamba	644***	64	40-144	0-40	0.50			
Pictoram	644	64	44-138	0-40	0.50			

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PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Alachlor (MS)	645	28	45-140	0-30	1.0
Butachtor	645	28	50-124	0-40	1.0
Diphenamid	645	28	57-119	0-40	1.0
Fluridone	645	28	45-154	0-40	1.0
Lethane	645	28	33-153	0-50	1.0
Norflurazon	645	28	48-110	0-40	1.0
Surrogate - Triphenylphosphate	645	28	40-125	NA .	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
Acenaphthene (MS)	8310	2	44-162	0-52	1.0
Acenaphthylene	8310	2	10-139	0-40	1.0
Acridine	8310***	2	10-200	0-40	0.50
Anthracene	8310	2	10-126	0-40	0.20
Benzo(a)anthracene	8310	2	12-135	0-40	0.20
Benzo(b)fluoranthene	8310	2	6-150	0-40	0.20
Benzo(k)fluoranthene	8310	2	10-159	0-40	0.50
Benzonitrile	8310***	2	10-200	0-40	10
Benzo(g,h,i)perylene	8310	2	10-120	0-40	0.50
Benzo(a)pyrene	8310	2	10-128	0-40	0.20
7,8-Benzoquinoline	8310***	2	10-200	0-40	1.0
Carbazole	8310***	2	10-150	0-40	1.0
Chrysene (MS)	8310	2	10-199	0-40	0.20
Dibenzo(a,h)anthracene	8310	2	10-110	0-40	1.0
2,4-Dimethylquinoline	8310***	2	10-200	0-40	20
Fluoranthene	8310	2	41-155	0-54	0.50
Fluorene (MS)	8310	2	10-142	0-40	0.50
Indeno(1,2,3-cd)pyrene	8310	2	10-116	0-40	0.50
1-Methylnaphthalene	8310	2	10-125	0-40	1.0
2-Hethylnaphthalene	8310	2	10-125	0-40	1.0
8-Methylquinoline	8310***	2	10-200	0-40	5.0
Naphthalene (MS)	8310	2	50-135	0-40	1.0
Phenanthrene	8310	2	10-155	0-40	0.20
Pyrene (MS)	8310	2	50-158	0-43	0.50
Quinaldine	8310***	2	10-200	0-40	5.0
Ouinoline	8310***	2	10-200	0-40	40
Surrogate - 2-Fluorobiphenyl	8310	2	60-140	NA.	NA
Surrogate - 4-Terphenyi-d4	8310	2	60-140	NA	NA

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOO (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/L)
Acetaldehyde	8315	2	30-110	0-40	200
Formaldehyde	8315	2	50-155	0-30	20
Aldicarb (Temik) (MS)	8318	2	34-124	0-40	10
Aldicarb sulfone	8318_	2	54-116	0-40	5.0
Aldicarb sulfoxide	8318***	2	30-140	0-40	5.0
Carbaryl (Sevin)	8318	2	55-125	0-40	5.0
Carbofuran (Furadan) (MS)	8318	2	52-125	0-40	10
Dioxacarb	8318	2	56-124	0-40	5.0
3-Hydroxycarbofuran	8318	2	47-123	0-40	5.0
Methiocarb (Mesurol)	8318	2	51-137	0-40	5.0
Methomyl (Lannate)	8318	2	57-125	0-40	5.0
Oxamyl (MS)	8318***	2	50-150	0-40	5.0
Promecarb	8318	2	48-122	0-40	5.0
Propoxur (Baygon)	8318	2	47-127	0-40	5.0

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TABLE 5.1. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY® (% Rec)	PRECISION® (% RPD)	PQL** (ug/L)
1,3-Dinitrobenzene (MS)	8330	2	54-166	0-30	0.20
2,4-Dinitrotoluene (MS)	8330	2	60-140	0-30	0.20
2,6-Dinitrotoluene	8330	2	60-140	0-30	0.50
Diphenylamine	8330***v	2	65-140	0-30	10
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDK)	8330	2	54-166	0-30	1.0
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)	8330	2	41-165	0-30	5.0
Nitrobenzene	8330	2	52-152	0-30	5.0
Nitroglycerin	8330***V	2	71-121	0-22	10
n-Nitrosodiphenylamine	8330***V	2	55-121	0-30	10
2-Nitrotoluene (MS)	8330	2	50-144	0-30	20
3-Witrotoluene	8330	2	55-165	0-30	10
4-Nitrotoluene	8330	2	54-166	0-30	20
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	8330	2	54-162	0-30	20
1,3,5-Trinitrobenzene	8330	2	50-150	0-30	0.50
2,4,6-Trinitrotoluene	8330	2	50-17	0-30	0.20
Surrogate - 2-Fluorobiphenyl	8330	2	40-140	NA	NA.

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (mg/kg)
Aluminum	6010(3050)	2	70-130	0-30	20
	CLP	45	70-130	0-20	40
Antimony (MS)	6010(3050)	2	70-130	0-30	5.0
,,	CLP	45	70-130	0-20	12
	7041(3050)	2	70-130	0-30	5.0
Arsenic (MS)	6010(3050)	2	70-130	0-30	10
Ar serie (ils)	7060(3050)	2	70-130	0-30	1.0
	7061	2	70-130	0-30	1.0
	CLP	45	70-130	0-20	20
Barium (MS)	6010(3050)	2	70-130	0-30	1.0
Bair (ms)	CLP	45	70-130	0-20	1.0
	/242.7252				
Beryllium (MS)	6010(3050)	2	70-130	0-30	0.50
	CLP 7091(3050)	45	70-130 70-130	0-20 0-30	1.0
	7091(3050)	2	70-130	0-30	0.10
Boron	6010(3050***)	2	70-130	0-30	5.0
Cadmium (MS)	6010(3050)	2	70-130	0-30	0.50
	CLP	45	70-130	0-20	1.0
	7131(3050)	2	70-130	0-30	0.10
Calcium	6010(3050)	2	70-130	0-30	50
Cataran	CLP	45	70-130	0-20	1000
	(040.7350)		70.470	0.70	1.0
Chromium (MS)	6010(3050)	45	70-130	0-30 0-20	1.0
	CLP 7191(3050)	2	70-130 70-130	0-30	1.0
Cobalt (MS)	6010(3050) CLP	45	70-130 70-130	0-30 0-20	1.0
6 (H0)	(010/7050)		70.470	0.70	3.5
Copper (MS)	6010(3050) CLP	45	70-130 70-130	0-30 0-20	2.5 5.0
	(010,7050)	3	70.470	0.70	F 0
Iron	6010(3050) CLP	2 45	70-130 70-130	0-30 0-20	20
					
Lead (MS)	6010(3050)	2	70-130	0-30	5.0
	7421(3050) CLP	2 45	70-130 70-130	0-30 0-20	0.50
					
Lithium	3500-Li B(3050***)	4/2	70-130	0-30	10
Magnesium	6010(3050)	2	70-130	0-30	50
	CLP	45	70-130	0-20	1000
Manganese (MS)	6010(3050)	2	70-130	0-30	1.0
nungaliese (Na)	CLP	45	70-130	0-20	3.0
Manager (MA)	7/71	,		0-30	
Mercury (MS)	7471	2 45	70-130 70-130	0-30	0.030
	CLP		70-130	0-20	0.030
Molybdenum	6010(3050)	. 2	70-130	0-30	1.0
Niekel (MC)	4010/70503	3	70-130	0-30	4.0
Nickel (MS)	6010(3050) CLP	2 45	70-130	0-30	8.0
Potassium	6010(3050)	2	70-130	0-30 0-20	100
	7610(3050)	45 2	70-130 70-130	0-30	1000

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (mg/kg)
Selenium (MS)	6010(3050)	2	70-130	0-30	50
	7740(3050)	2	70-130	0-30	1.0
	7741	2	70-130	0-30	1.0
	CLP	45	70-130	0-20	1.0
Silica	6010(3050***)	2	70-130	0-30	50
Silver (MS)	6010(3050)	2	70-130	0-30	1.0
	CLP	45	70-130	0-20	2.0
	7761(3050)	2	70-130	0-30	0.10
Sodium	6010(3050)	2	70-130	0-30	50
	CLP	45	70-130	0-20	1000
Strontium	6010***(3050***)	2	70-130	0-30	1.0
Thattium (MS)	6010(3050)	2	70-130	0-30	50
	7841(3050)	2	70-130	0-30	1.0
	CLP	45	70-130	0 -20	2.0
Tin	6010***V(3050***V)	2	70-130	0-30	5.0
Titanium	6010***(3050***)	2	70-130	0-30	1.0
Tributyl tin	Atomic absorption	40	70-130	0-40	0.10
Vanadium (MS)	6010(3050)		70-130	0-30	1.0
	CLP	45	70-130	0-20	10
Zinc (MS)	6010(3050)	2	70-130	0-30	2.0
	CLP	45	70-130	0-20	4.0
Zinc phosphide	FDER Special Method	31	NA	NA	NA
Zirconium	6010***(3050***)	2	70-130	0-30	500

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (mg/kg)				
Ammonia (as N) (extractable)	EPA-CE-3-140 350.3(EPA-CE)	46 3/46	75-125 75-125	0-30 0-30	0.50 0.50				
800	EPA-CE	46	60-140	0-40	200				
вти	D240-76	38	70-130	0-30	200 BTU/1b				
Carbon, total organic	EPA-CE [Walkley- Black]	46 [43]	60-140	0-40	50				
	9060	2	60-140	0-40	50				
Cation exchange capacity	9081/EPA-CE	2/46	70-130	0-40	0.0033 meg/100 g				
Chloride (extractable)	9251(5050)	2	75-125	0-30	20				
	9252 407A	2 4	75-125 75-125	0-30 0-30	20 20				
Chioride, total	9251(5050) 9056(5050)	2 2	70-130 70-130	0-40 0-40	100				
COO		46							
Coliform, fecal	908C(AOAC)	4(36)	60-140 NA	0-40 NA	100 3 MPN/g				
Coliform, total	908A(A0AC)	4(36)	NA NA	NA NA	3 MPN/g				
Cyanide, amenable to chlorination	9012 9010	2 2	NA NA	0-50 0-40	1.0				
Cyanide, reactive	7.3.3.2	2	NA	0-50	1.0				
Cyanide, total	9012(9010A) 9010 CLP	2 2 45	75 - 125 75 - 125 85 - 115	0-30 0-30 0-25	1.0 1.0 0.30				
EP Toxicity	1310	2	NA	NA NA	NA				
Formal dehyde	HZOIN	35	80-120	0-20	13				
Fluoride (extractable)	340.2	3	75-125	0-25	4.0				
Halogens, total	9056(5050)	2	70-130	0-40	200				
Halogens, total organic (EOX)	EPA-600/4-84-008	44	60-140	0-50	10				
Hydrogen ion (pH)	9045	2	NA	0-10	на				
ignitability	1010	2	NA	NA NA	NA				
Nitrate (as N) (extractable)	EPA-CE	46	75-125	0-30	5.0				
Nitrate-Nitrite (as N)	EPA-CE	46	75-125	0-30	5.0				
Nitrite (as N) (extractable)	EPA-CE	46	75-125	0-30	5.0				
Nitrogen, organic	EPA-CE	46	NA	NA.	25				
Nitrogen, total	EPA-CE	46	NA	NA.	30				
Nitrogen, total Kjeldahl	EPA-CE	46	65-135	0-30	25				

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	pQL±± (mg/kg)
Oil and Grease	9070(9071)	2	60-140	0-50	10
Orthophosphate	365.1	3	75-125	0-30	1.0
Paint filter liquids	9095	2	NA NA	0-40	NA
Petroleum hydrocarbons	9073	2	60-140	0-50	10
Phenolics, total recoverable	9066(EPA-CE) 9065-(EPA-CE)	2 (46) 2 (46)	60-140 60-140	0-40 0-40	0.40
Phosphorus, total	EPA-CE-3-2/3 EPA-CE-3-2/2	46 46	60-140 60-140	0-40 0-40	25 5.0
Radioactivity, alpha	900.0/9310	2	NA	0-30	NA
Radioactivity, beta	900.0/9310	2	. NA	0-30	NAAK
Residue, fixed (% ash)	EPA-CE	46	NA_	0-40	0.10%
Solids, total	EPA-CE	46	NA	0-30	0.10%
Solids, volatile	EPA-CE	46	75-125	0-30	0.10%
Specific gravity	EPA-CE	46	. NA	0-10	NA
Streptococcus, fecal	910A	4/36	NA NA	NA.	3 MPN/g
Sulfate (extractable)	9036 9038 375.3	2 2 3	75-125 75-125 75-125	0-25 0-25 0-25	100 100 10
Sulfide	9030-SL	2	50-150	0-50	10
Sulfide, reactive	7.3.4.2	2	NA	0-50	10
Sutfur	D129-64/9056(5050)	38/2	70-130	0-30	170
Surfactants (MBAS) (extractable)	425.1	3	60-140	0-40	20
Toxic compound leaching procedure	1311	48	NA .	NA	NA
Water (Karl Fisher)	D1744	38	NA	0-30	50

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Benzyl chloride	8010(5030)	2	50-150	0-30	5.0
Bromobenzene	8010(5030)	2	70-130	0-30	50
Bromodichloromethane	8010(5030)	2	42-172	0-30	5.0
Bromoform	8010(5030)	2	13-159	0-30	25
Bromomethane	8010(\$030)	2	10-144	0-30	5.0
Carbon tetrachloride	8010(5030)	2	43-143	0-30	5.0
Chlorobenzene (MS)	8010(5030)	2	31-122	0-27	5.0
Chloroethane	8010(5030)	2	46-137	0-30	5.0
Chloroform	8010(5030)	2	49-133	0-30	5.0
1-Chlorohexane	8010(5030)	2	50-150	0-30	5.0
2-Chloroethylvinyl ether	8010(5030)	2	14-186	0-80	50
Chloromethane	8010(5030)	2	10-193	0-30	5.0
Chlorotoluenes	8010(5030)	2	70-130	0-30	50
Dibromochloromethane	8010(5030)	2	24-191	0-30	5.0
Dibromomethane	8010(5030)	2	70-130	0-30	25
1,2-Dichlorobenzene	8010(5030)	2	10-208	0-30	5.0
1,3-Dichlorobenzene	8010(5030)	2	10-187	0-30	5.0
1,4-Dichlorobenzene	8010(5030)	2	42-143	0-30	5.0
Dichlorodifluoromethane	8010(5030)	2	70-130	0-30	5.0
1,1-Dichloroethane	8010(5030)	2	47-132	0-30	5.0
1,2-Dichloroethane	8010(5030)	2	51-147	0-30	5.0
1,1-Dichloroethylene (MS)	8010(5030)	2	51 -13 2	0-28	5.0
cis/trans 1,2-Dichloroethene	8010(5030)	2	38-155	0-30	5.0
Dichloromethane (Methylene chloride)	8010(5030)	2	25-162	0-30	5.0
1,2-Dichloropropane	8010(5030)	2	44-156	0-30	5.0
cis/trans-1,3-Dichloropropylene	8010(\$030)	2	22-178	0-30	5.0
1,1,2,2-Tetrachloroethane	8010(5030)	2	10-184	0-30	5.0
1,1,1,2-Tetrachtoroethane	8010(5030)	2	70+130	0-30	5.0
Tetrachloroethene	8010(5030)	2	26-162	0-30	5.0
1,1,1-Trichloroethane	8010(5030)	2	41-138	0-30	5.0
1,1,2-Trichloroethane	8010(5030)	2	39-136	0-30	5.0
Trichloroethene (MS)	8010(5030)	2	56-133	0-26	5.0
Trichlorofluoromethane	8010(5030)	2	21-156	0-30	5.0

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	HETHOD (Prep)	REFERÊNCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)		
1,2,3-Trichloropropane	8010(5030)	_2	50-150	0+30	5.0		
Vinyl chloride	8010(5030)	2	28-163	0-30	5.0		
1,2-Dibromoethane (EDB)	8010***(5030)	2	75+125	0-30	5.0		
Surrogate - Bromochloromethane	8010(5030)	2	43-127	NA_	NA.		

^{&#}x27; EDB determined on Hall detector with PQL of 5.0 ug/kg at client's request.

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	HETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acetone	8015***(5030)	2	40-130	0-30	130
2-Butanone (MEK)	8015(5030)	2	60-130	0-40	130
Diethyl ether	8015(5030)	2	10-130	0-50	130
Ethanol	8015(5030)	2	20-140	0-45	5000
Ethyl methacrylate	8015***(5030)	2	42-125	0-40	50
Isobutanol	8015***(5030)	2	50-120	0-40	5000
Isopropanol	8015***(5030)	2	30-140	0-40	5000
Methacrylonitrile	8015***(5030)	2	10-140	0-60	500
Methanol	8015***(5030)	2	50-150	0-40	5000
Methyl methacrylate	8015***(5030)	2	45-132	0-42	50_
4-Methyl-2-pentanone (MIBK)	8015 (5030)	2	65-125	0-40	130
Methyl t-butyl ether (MTBE)	8015***(5030)	2	50-150	0-30	50
Propionitrile	8015***(5030)	2	10-130	0-50	500
Gasoline	8015 (modified)	12	40-140	0-40	250
Methanol (MS)	8015 (modified/DEI*)	2	50-1 <u>5</u> 0	0-50	1000
Ethanol	8015 (modified/DEI*)	2	50-150	0-50	1000
n-Propanol	8015 (modified/DEI*)	2	50-150	0-50	1000
Isopropanol (MS)	8015 (modified/DEI*)	2	50-150	0-50	1000
n-Butanol	8015 (modified/DEI*)	2	50-150	0-50	1000
Isobutanol	8015 (modified/DEI*)	2	50-150	0-50	1000_
Ethylene glycol (MS)	8015 (modified/DEI*)	2	50-150	0-50	10000
Diethylene glycol	8015 (modified/DEI*)	2	50-150	0-50	10000
Propylene glycol	8015 (modified/DEI*)	2	50-150	0-50	10000
Triethylene glycol	8015 (modified/DEI*)	2	50-150	0-50	10000
Tetraethylene glycol	8015 (modified/DEI*)	2	50-150	0-50	25000

^{*} DEI = Direct Extract Injection

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	HETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Benzene (MS)	8020(5030)	2	63-133	0-27	5.0
Chlorobenzene (MS)	8020(5030)	2	69-129	0-25	5.0
1,2-Dichlorobenzene	8020(5030)	2	37-154	0-30	5.0
1,3-Dichlorobenzene	8020(5030)	2	50-141	0-30	5.0
1,4-Dichlorobenzene	8020(5030)	2	42-143	0-30	5.0
Ethylbenzene	8020(5030)	2	32-160	0-30	5.0
Methyl tert-butyl ether (MTBE)	8020***(5030)	2	50-150	0-30	50
Toluene (MS)	8020(5030)	2	70-138	0-26	5.0
Xylenes	8020(5030)	2	50-150	0-30	5.0
m-Xylene	8020(5030)	2	50-150	0-30	5.0
o+p Xylene	8020(5030)	2	50-150	0-30	5.0
Surrogate - a,a,a-Trifluorotoluene	8020(5030)	2	67-137	NA NA	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acrolein	8030(5030)	2	88-118	0-30	1000
Acrylonitrile	8030(5030)	2	71-135	0-30	500
Acetonitrile .	8030***(5030)	2	20-115	0-30	5000

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL++ (ug/kg)
2-Chlorophenol (MS)	8040(3550)	2	27-150	0-26	330
4-Chloro-3-methylphenol (MS)	8040(3550)	2	20-151	0-39	330
2,4-Dichlorophenol	8040(3550)	2	44-119	0-40	330
2,4-Dimethylphenol	8040(3550)	2	24-118	0-40	330
2,4-Dinitrophenol	8040(3550)	2	12-145	0-65	1700
2-Methyl-4,6-dinitrophenol	8040(3550)	2	30-136	0-40	1700
3-Methyl phenol (m-cresol)	***8040(3550)	2	10-150	0-50	330
2-Methyl phenol (o-cresol)	***8040(3550)	2	10-150	0-50	330
4-Methyl phenol (p-cresol)	***8040(3550)	2	10-150	0-50	330
Cresols	8040(3550)	2	10-150	0-50	330
2-Nitrophenol	8040(3550)	2	43-117	0-40	330
4-Nitrophenol (MS)	8040(3550)	2	10-130	0-34	1700
Pentachlorophenol (MS)	8040(3550)	2	10-162	0-80	1700
Phenol (MS)	8040(3550)	2	13-149	0-30	330
Trichlorophenols	8040(3550)	2	NA.	NA	330
2,3,4,6-Tetrachlorophenol	***8040(3550)	2	50-150	0-40	660
2,3,4,5-Tetrachlorophenol	***8040(3550)	2	50-150	0-40	660
Tetrachtorophenols	8040(3550)	2	NA .	NA	660
2,4,6-Trichlorophenol	8040(3550)	2	53-119	0-40	330
2,4,5-Trichlorophenol	***8040(3550)	2	53-119	0-40	330
Surrogate - 2,4,6-Tribromophenol	8040(3550)	2	10-186	NA.	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Butyl benzyl phthalate (MS)	8060(3550)	2	10-137	0-66	330
Bis(2-ethylhexyl) phthalate (MS)	8060(3550)	2	10-151	0-54	330
Di-n-butyl phthalate (MS)	8060(3550)	2	14-123	0-41	330
Diethyl phthalate (MS)	8060(3550)	2	10-145	0-34	330
Dimethyl phthalate (MS)	8060(3550)	2	10-147	0-31	330
Di-n-octyl phthalate (MS)	8060(3550)	2	10-147	0-86	330
Surrogate - 2-fluorobiphenyl	8060(3550)	2	17-164	AK	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	HETHCO (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Aldrin (MS)	8080(3550) CLP-2/88	2 6	40-137 34-132	0-39 0-43	1.7
	CLP-3/90	62	34-132	0-43	1.7
Benfluratin	8080***(3550)	2	40-140	0-40	0.33
alpha-BHC	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	37-134	0-40	1.7
beta-BHC	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	17-147	0-40	1.7
gamma-BHC (Lindane) (MS)	8080(3550) CLP-2/88 CLP-3/90	2 6 62	41-134 46-127 46-127	0-36 0-50 0-50	1.7 8.0 1.7
delta-BHC	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	19-140	0-40	1.7 8.0
Carbophenothion	8080***(3550)	2	20-150	0-50	33
Chlordane	8080(3550)	2	45-119	0-40	17
alpha Chlordane	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	45-140	0-40	1.7 80
gamma Chlordane	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	45-140	0-40	1.7 80
Chlorobenzilate	8081***V(3550)	2	50-150	0-40	17
Chloroneb	8080***V(3550)	2	49-125	0-30	13
Chloropropylate	8080***V(3550)	2	51-125	0-30	16
Chlorothalonil	8080***(3550)	2	<u>35-1</u> 30	0-40	6.7
4,4/-DDD	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	31-141	0-50	3.3 16
4,4'-DDE	8080(3550)/CLP-3/90 CLP-2/88	2/62	30-145	0-50	3.3 16
4,4'-DDT (MS)	8080(3550) CLP-2/88 CLP-3/90	2 6 62	48-150 23-134 23-134	0-34 0-50 0-50	3.3 16 3.3
Dicofol (Kelthane)	8081***V(3550)	2	40-125	0-40	20
Dieldrin (MS)	8080(3550) CLP-2/88 CLP-3/90	2 6 62	42-139 31-134 31-134	0-41 0-38 0-38	3.3 16 3.3
Endosulfan I	8080(3550)/CLP-3/90 CLP-2/88	2/62	45-153	0-40	1.7 8.0
Endosulfan II	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	10-202	0-65	3.3 16
Endosulfan sulfate	8080(3550)/CLP-3/90 CLP-2/88	2/6 2 6	26-144	0-50	3.3 16
Endrin (MS)	8080(3550) CLP-2/88 CLP-3/90	2 6 62	44-151 42-139 42-139	0-31 0-45 0-45	3.3 16 3.3
Endrin aldehyde	8080(3550)/CLP-3/90	2/62	10-150	0-50	3.3
Endrin ketone	CLP-2/88 CLP-3/90	6 62	NA NA	NA AK	16 3.3
Etridiazole	8080***V(3550)	2	50-125	0-30	0.33
Heptachlor (MS)	8080(3550) CLP-2/88 CLP-3/90	2 6 62	40-136 35-130 35-130	0-34 0-31 0-31	1.7 8.0 1.7

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Heptachlor epoxide	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	37-142	0-40	1.7 8.0
Isodrin	8081***V(3550)	2	10-150	0-50	3.3
Kepone	8081***V(3550)	2_	10-150	0-50	17
Methoxychlor	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	50-140	0-40	17 80
Hirex	8081***V(3550)	2	20-100	0-50	33
Pendimethalin	8080***(3550)	2	35-125	0-50	67
Permethrin (total)	8080***(3550)	2	40-140	0-50	33
Propachlor	8080***V(3550)	2	51-125	0-30	16
Toxaphene	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	41-126	0-50	170 160
Trifluratin	8080***(3550)	2	40-140	0-40	0.33
PCB-1016	8080(3550)/CLP-3/90 CLP-2/88	2/62 6	69-107	0-21	33 80
	EPA-600/4-81-045	61	50-130	0-50	5000
PCB-1221	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	15-178 50-130	0-20	67 80 5000
PCB-1232	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	10-215 50-130	0-20 0-50	33 80 5000
PCB-1242	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	39-150 50-130	0-20 0-50	33 80 5000
PCB-1248	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	38-158 50-130	0-20 0-50	33 80 5000
PCB-1254	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	66-122 50-130	0-23 0-50	33 160 5000
PCB-1260	8080(3550)/CLP-3/90 CLP-2/88 EPA-600/4-81-045	2/62 6 61	58-122 50-130	0-20	33 160 5000
Surrogate - Dibutylchlorendate (DBC)	8080(3550) CLP-2/88	2 6	45-131 24-150	NA NA	NA NA
Surrogate - 2,4,5,6-Tetrachioro-m- xylene (TCHX)	8080(3550) CLP-3/90	2 62	19-132 60-150	NA NA	NA NA
Surrogate - Decachlorobiphenyl (DCB)	8080(3550) CLP-3/90	2 62	47-126 60-150	NA NA	NA NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPO)	PQL=+ (ug/kg)
2,4-Dinitrotoluene (M\$)	8090(3550)(FID) (ECD)	2	10-125 10-125	0-40 0-40	330 10
2,6-Dinitrotoluene (MS)	8090(3550)(F1D) (ECD)	2	10-126 10-126	0-40 0-40	330 10
Isophorone (MS)	8090(3550)	2	10-117	0-40	330
Nitrobenzene (MS)	8090(3550)	2	10-118	0-40	330
Surrogate - 2-Fluorobiphenyl	8090(3550)(F1D)	2	17-164	NA	NA.
Surrogate - 2,4,5,6-Tetrachioro-m-xylene (TCMX)	8090(3550)(ECD)	2	19-132	NA	МА

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acenaphthene	8100(3550)	2	37-115	0-32	330
Acenaphthylene	8100(3550)	2	36-114	0-32	330
Benzo(a)pyrene .	8100(3550)	_2	21-125	0-45	330
Benzo(b+k)fluoranthene	8100(3550)	2	26-128	0-41	330
Benzo(g,h,i)perylene	8100(3550)	2	25-126	0-42	330
Carbazole	8100***(3550)	2	16-132	0-31	330
Chrysene+Benzo(a)anthracene	8100(3550)	2	30-127	0-42	330
Fluoranthene	8100(3550)	2	28-132	0-33	330
Fluorene	8100(3550)	2	36-117	0-33	330
Indeno(1,2,3-cd)Pyrene + Dibenzo(a,h)anthracene	8100(3550)	2	20-131	0-47	330
1-Methylnaphthalene	8100(3550)	2	20-140	0-50	330
2-Methylnaphthalene	8100(3550)	2	20-140	0-50	330
Napthalene	8100(3550)	2	29-111	0-45	330
Phenanthrene + Anthracene	8100(3550)	2	38-118	0-32	330
Pyrene	8100(3550)	2	35-123	0-32	330
Diesel	8100 (modified)	12	40-140	0-40	10000
Mineral spirits	8100 (modified)	12	40-140	0-40	10000
Surrogate - 2-Fluorobiphenyl	8100(3550)	2	17-164	NA	NA
Surrogate - Decafluorobiphenyl	8100 (modified) (3550)	12	20-150	NA.	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acephate	8141***(3550)	2	40-140	0-50	167
Alachior	8141***(3550)	2	45-140	0-30	33
Ametryn	8141***V(3550)	2	40-140	0-50	66
Atrazine	8141***V(3550)	2	40-125	0-30	66
Azinphos methyl (MS)	8141(3550)	2	16-129	0-50	66
Bolstar (MS)	8141(3550)	2	58-156	0-40	33
Bromacil	8141***(3550)	2	40-140	0-50	66
Butylate	8141***(3550)	2	38-145	0-76	66
Carbophenothion	8141***V(3550)	2	20-150	0-40	66
Chlorpyrifos	8141(3550)	2	82-115	0-40	33
Coumaphos	8141(3550)	2	51-147	0-40	330
Cycloate	8141***(3550)	2	46-159	0-47	66
Demeton-0	8141(3550)	2	36-120	0-40	83
Demeton-S	8141(3550)	2	36-120	0-40	83
Diazinon (MS)	8141(3550)	2	36-124	0-30	33
Dichlofenthion	8141***V(3550)	2	40-140	0-50	33
Dichlorvos	8141(3550)	2	49-120	0-40	66
Dimethoate	8141(3550)	2	38-120	0-40	2000
Disulfoton (MS)	8141(3550)	2	10-134	0-93	130
Dioxathion	8141***V(3550)	2	40-140	0-50	330
EPN	8141(3550)	2	48-124	0-30	33
EPTC	8141***(3550)	2	46-154	0-55	66
Ethion	8141(3550)	2	40-138	0-40	17
Ethoprop	8141(3550)	2	58-113	0-40	17
Famphur	8141(3550)	2	10-129	0-60	330
Fenamiphos	8141***(3550)	2	40-160	0-40	17
Fensul fothion	8141(3550)	2	43-145	0-40	330
Fenthion	8141(3550)	2	10-128	0-60	33
Fonofos	8141***(3550)	2	40-160	0-40	33
Hexazinone	8141***(3550)	2	40-140	0-50	33
Isofenphos	8141***(3550)	2	40-160	0-40	17
Malathion	8141(3550)	2	60-140	0-40	33
Merphos	8141(3550)	2	50-130	0-40	33
Metalaxyl	8141***(3550)	2	40-140	0-50	33
He tham i dophos	8141***(3550)	2	40-140	0-50	66
Methyl chlorpyrifos	8141***(3550)	2	40-140	0-50	33
Metolachior	8141***(3550)	2	40-140	0-50	33
Metribuzin	8141***(3550)	. 2	40-140	0-50	33
Mevinonos	8141(3550)	2	34-125	0-40	66
Molinate	8141***(3550)	2	37-127	0-74	66
Monocrotophos	8141***(3550)	2	40-140	0-50	330
Nated	8141(3550)	2	54-102	0-40	330

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/kg)
Norflurazon	8141***(3550)	2	40-140	0-50	33
Parathion, ethyl (MS)	8141 (3550)	2	15-141	0-79	33
Parathion, methyl (MS)	8141(3550)	2	40-104	0-40	17
Pebulate	8141***(3550)	2	22-172	0-50	33
Phorate	8141(3550)	2	36-125	0-40	33
Prometon	8141***V(3550)	2	40-140	0-50	66
Prometryn	8141***V(3550)	2	40-140	0-50	66
Propazine	8141***V(3550)	2	40-140	0-50	66
Ronnel	8141(3550)	2	22-127	0-35	33
Simazine	8141***V(3550)	2	20-150	0-50	66
Stirophos (Tetrachlorvinphos)	8141(3550)	2	48-125	0-40	33
Sulfotepp (MS)	8141(3550)	2	13-171	0-65	17
Terbufos	8141***(3550)	2	40-140	0-50	17
Terbutryn	8141***V(3550)	2	40-140	0-50	330
Terbutylazine	8141***V(3550)	2	40-140	0-50	66
Thionazin	8141(3550)	2	25-160	0-60	66
Tokuthion (Prothiofos)	8141(3550)	2	44-125	0-40	33
Triadimefon	8141***(3550)	2	40-140	0-50	33
Trichloronate	8141(3550)	2	49-161	0-40	330
Vernolate	8141***(3550)	2	39-147	0-45	66
Surrogate - Ronnel	8141(3550)	2	22-127	NA	HA
Surrogate - Tokuthion	8141(3550)	2	50-1 25	NA	NA
Surrogate - Triphenylphosphate	8141(3550)	2	40-125	NA NA	HA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/kg)
2,4-D (MS)	8150	2	10-130	0-47	_ 50
Dalapon	8150	2	10-170	0-40	2000
2,4-DB	8150	2	20-160	0-40	50
Dicamba	8150	2	20-160	0-40	20
Dichlorprop	8150	2	30-170	0-40	100
Dinoseb	8150	2	30-170	0-40	100
МСРА	8150	2	30-170	0-40	2000
МСРР	8150	2_	30-170	0-40	2000
Picloram	8150***	2	10-150	0-40	3.3
2,4,5-T (MS)	8150	2	24-115	0-46	50
2,4,5-TP (Silvex) (MS)	8150	2	10-150	0-54	50
Surrogate - 2,4-Dichlorophenoxy butanoic acid (2,4-DB)	8150	2	20-160	NA	NA
Surrogate - 2,4-Dichlorophenyl acetic acid (DCAA)	8150	2	10-148	NA	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acetone	8240(5030)/8260(5030) CLP-2/88; 3/90	2 6/62	29-92	0-40	50
Acetonitrile	82408(5030)	2	78-151	0-40	200
Acrolein	8240(5030)/8260A(5030)	2	22-164	0-65	200
Acrylonitrile	8240(5030)/8260A(5030)	2	61-145	0-40	100
Benzene (MS)	8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	2 6 62	48-150 66-142 66-142	0-27 0-21 0-21	5.0 5.0 10
Benzyl Chloride	8240B(5030)	2	50-150	0-40	100
Bromobenzene	8260A(5030)	2	50-150	0-40	10
Bromodichloromethane	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	35-155	0-40	5.0
	CLP-3/90_	62		<u> </u>	10
Bromoform	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 6 2	45-16 9	0-40	5.0 10
Bromomethane (Methyl bromide)	8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	2/6/62	10-242	0-65	10
2-Butanone (MEK)	8240(5030)/8260(5030)/ 8260A(5030)	2	10-111	0-40	50
	CLP-2/88; 3/90	6/62			10
n-Butylbenzene	8260A(5030)	2	50-150	0-40	5.0
sec-Butylbenzene	8260A(5030)	. 2	50-150	0-40	5.0
tert-Butylbenzene	8260A(5030)	2	50-150	0-40	5.0
Carbon disulfide	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 62	35-244	0-65	5.0
Carbon tetrachloride	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6	70-140	0-40	5.0
Chlorobenzene (MS)	8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	2 6 62	54-138 60-133 60-133	0-33 0-21 0-21	5.0 5.0 10
2-Chloro-1,3-butadiene (Chloroprene)	8240B(5030)	2	28-256	0-65	10
Chloroethane	8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	2/6/62	44-136	0-40	10
2-Chloroethyl vinyl ether	8240(5030)/8260A(5030)	2	10-305	0-65	50
Chloroform	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	51-138	0-40	5.0
Chloromethane	CLP-3/90 8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	2/6/62	10-273	0-65	10
Chloropicrin	8240***(5030)	2	50-130	0-40	5.0
3-Chloropropene (Allyl chloride)	82408(5030)	2	88-127	0-40	5.0
2-Chlorotoluene	8240***(5030)/8260A(5030)	2	48-125	0-40	5.0
4-Chlorotoluene	8260A(5030)	2	50-150	0-40	5.0
Dibromochloromethane	8240/CLP-2/88/8260A(5030) CLP-3/90	2/6 62	53-149	0-40	5.0 10

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
1,2-Dibromo-3-chloropropane (DBCP)	8240B(5030)/8260A(5030)	2	26-165	0-40	10
1,2-Dibromoethane (EDB)	8240B(5030)/8260A(5030)	2	86-153	0-40	5.0
Dibromomethane	8240B(5030)/8260A(5030)	2	<u>5</u> 0-150	0-40	5.0
1,2-Dichlorobenzene	8240(5030)/8260A(5030)	2	81-113	0-40	5.0
1,3-Dichlorobenzene	8240(5030)/8260A(5030)	2	63-130	0-40	5.0
1,4-Dichlorobenzene	8240(5030)/8260A(5030)	2	69-126	0-40	5.0
trans-1,4-Dichloro-2-butene	8240B(5030)	2	79-178	0-40	5.0
Dichlorodifluoromethane	8240B(5030)/8260A(5030)	2	50-150	8-40	5.0
1,1-Dichloroethane	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	59-155	0-40	5.0
	CLP - 3/0	62			10
1,2-Dichloroethane	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 62	49-155	0-40	5.0 10
cis/trans-1,2-Dichloroethene	8240(5030)/CLP-2/88/	2/6	54-156	0-40	5.0
cray ti ara-1,2 vicintor vernene	8260A(5030) CLP-3/90	62	34 130	0-40	10
1,1-Dichloroethene (MS)	8240(5030)/8260A(5030)	2	36-161	D-50	5.0
	CLP-2/88 CLP-3/90	6 62	59-172 59-172	0-22 0-22	5.0 10
1,2-Dichloropropane	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	10-210	0-65	5.0
	CLP-3/90	62			10
1,3-Dichloropropane	8260A(5030)	2	50-150	0-40	5.0
2,2-Dichloropropane	8260A(5030)	2	50-150	0-40	5.0
1,1-Dichloropropene	8260A(5030)	2	50-150	0-40	5.0
cis-1,3-Dichloropropene	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	10-227	0-65	5.0
1.7.05-1-1	8240(5030)/CLP-2/88/	62	47.407		10
trans-1,3-Dichloroprop e ne	8260A(5030) CLP-3/90	2/6 62	17-183	0-65	5.0 10
Ethanol	8240(5030)	2	40-160	0-40	1000
Ethylbenzene	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	37-162	0-40	5.0
	CLP-3/90	62			10
Ethyl methacrylate	8240(5030)	2	47-87	0-40	5.0
Hexachlorobutadiene	8260A(5030)	2	50-150	0-40	5.0
2-Hexanone	8240(5030)/8260(5030) CLP-2/88; 3/90	2 6/62	22-86	0-40	50 10
Iodomethane	8240B(5030)/8260A(5030)	2	77-105	0-40	5.0
Isobutyl alcohol	82408(5030)	2	63-173	0-40	1000
I sopropy l benzene	8260A(5030)	2	50-150	0-40	5.0
p-lsopropyltoluene	8260A(5030)	2	50-150	0-40	5.0
Methacrylonitrile	82408 (5030)	2	69-145	0-60	100
Methylene chloride	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	10-221	0-65	5.0
	CLP-3/90	62			10

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Methylisothiocyanate	8240***(5030)	2	20-120	0-60	100
Methyl methacrylate	8240B(5030)	2	32-118	0-45	5.0
4-Methyl-2-pentanone (MIBK)	8240(5030)/8260A(5030) CLP-2/88; 3/90	2 6/62	64-125	0-49	50 10
Methyl t-butyl ether (MTBE)	8240***(5030)	2	40-150	0-40	50
Naphthalene	8260A(5030)	. 2	50-150	0-40	5.0
Pentach i oroethane	82408(5030)	2	41-165	0-50	25
Propionitrile (ethylcyanide)	82408(5030)	2	73-227	0-65	200
n-Propylbenzene	8260A(5030)	2	50-150	0-40	5.0
Styrene	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	89-101	0-40	5.0
 	CLP-3/90	62			10
1,1,1,2-TetrachLoroethane	8240B(5030)/8260A(5030)	2	50-150	0-40	5.0
1,1,2,2-Tetrachloroethane	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 62	46-157	0-40	5.0 10
Tetrachloroethene	8240(5030)/CLP-2/88/	2/6	64-148	0-40	5.0
	8260A(5030) CLP-3/90	62	57 140	0-40	10
Toluene (MS)	8240(5030)/8260A(5030)	2	51-141	0-27	5.0
	CLP-2/88 CLP-3/90	6 62	59-139 59-139	0-21 0-21	5.0 10
1,2,3-Trichlorobenzene	8260A(5030)	2	50-150	0-40	5.0
1,2,4-Trichlorobenzene	8260A(5030)	2	50-150	0-40	5.0
		2/6	52-162		
1,1,1-Trichloroethane	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	62	32-162	0-40	5.0 10
1,1,2-Trichloroethane	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 62	52-150	0-40	5.0 10
Trichloroethene (MS)	8240(5030)/8260A(5030)	2	43-140	0-27	5.0
Tremoroethere (AS)	CLP-2/88	6	62-137	0-24	5.0
	CLP-3/90	62	62-137	0-24	10.0
Trichlorofluoromethane	8240(5030)/8260A(5030)	22	17-181	0-65	5.0
1,2,3-Trichloropropane	8240(5030)/8260A(5030)	2	43-105	0-40	5.0
Trichlorotrifluoroethane	8240***(5030)	2	60-140	0-40	5.0
1,2,4-Trimethylbenzene	8260A(5030)	2	50-150	0-40	5.0
1,3,5-Trimethylbenzene	8260A(5030)	2	50-150	0-40	5.0
Vinyl acetate	8240(5030)/CLP-2/88/ 8260A(5030)	2/6	50-150	0-40	10
Vinyt chloride	8240(5030)/CLP-2/88; 3/90/ 8260A(5030)	2/6/62	10-251	0-65	10
Xylenes	8240(5030)/CLP-2/88/ 8260A(5030) CLP-3/90	2/6 62	50-150	0-40	5.0 10
Surrogate - Toluene-d8	8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	2 6 62	68-123 81-117 84-138	NA NA NA	MA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Surrogate - p-Bromofluorobenzene	8240(5030)/8260A(5030) CLP-2/88 CLP-3/90	2 6 62	64-126 74-121 59-113	NA NA NA	NA
Surrogate - Dibromofluoromethane	8260A(5030)	2	80-120	NA	NA
Surrogate - 1,2-Dichloroethane-d4	8240(5030) CLP-2/88; 3/90	2 6/62	46-143 70-121	NA HA	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPO)	PQL** (ug/kg)
Acenaphthene (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	51-108 31-137	0-26 0-19	330
Acenaphthylene	8270(3550)/CLP-2/88; 3/90	2/6/62	54-140	0-24	330
Acetophenone	8270(3550)	2	10-150	0-50	330
2-Acetylaminofluorene	8270(3550)	2	25-150	0-50	330
Aldrin	8270(3550)	2	10-166	0-40	330
4-Aminobiphenyl	8270(3550)	2	10-150	0-50	330
Aniline	8270(3550)	2	10-150	0-50	330
Anthracene	8270(3550)/CLP-2/88; 3/90	2/6/62	48-130	0-30	330
Aramite	8270(3550)	2	40-150	0-50	330
Benzidine	8270(3550)	2	10-200	0-100	2700
Benzo(a)anthracene	8270(3550)/CLP-2/88; 3/90	2/6/62	42-143	0-25	330
Benzoic acid	8270(3550)/CLP-2/88	2/6	10-150	0-50	1700
Benzo(b)fluoranthene	8270(3550)/CLP-2/88; 3/90	2/6/62	49-123	0-25	330
Benzo(k)fluoranthene	8270(3550)/CLP-2/88; 3/90	2/6/62	24-137	0-38	330
Benzo(g,h,i)perylene	8270(3550)/CLP-2/88; 3/90	2/6/62	10-219	0-50	330
8enzo(a)pyrene	8270(3550)/CLP-2/88; 3/90	2/6/62	44-141	0-29	330
Benzyl alcohol	8270(3550)/CLP-2/88	2/6	10-150	0-50	330
Benzyl chloride	8270B***(3550)	2	10-150	0-50	330
alpha-BHC	8270(3550)	2	10-150	0-50	330
beta-BHC	8270(3550)	2	24-149	0-40	330
del ta-BHC	8270(3550)	2	10-110	0-40	330
gamma-BHC	8270(3550)	2	10-150	0-50	330
Bis(2-chloroethoxy) methane	8270(3550)/CLP-2/88; 3/90	2/6/62	33-184	0-50	330
Bis(2-chloroethyl) ether	8270(3550)/CLP-2/88; 3/90	2/6/62	12-158	0-50	330
Bis(2-chloroisopropyl) ether	8270(3550)/CLP-2/88; 3/90	2/6/62	36-166	0-50	330
Bis(2-ethylhexyl) phthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-158	0-40	330
4-Bromophenyl phenyl ether	8270(3550)/CLP-2/88; 3/90	2/6/62	53-127	0-40	330
Butyl benzyl phthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-152	0-40	330
Carbazole	8270***(3550)/CLP-3/90	2/62	10-150	0-50	330
Technical Chlordane	8270(3550)	2	10-150	0-50	1700
p-Chloroaniline	8270(3550) CLP-2/88; 3/90	2 6/62	10-150	0-50	660 330
4-Chloro-3-methylphenol (MS) (p-Chloro-m-cresol)	8270(3550) CLP-2/88; 3/90	2 6/62	38-112 26-103	0-23 0-33	330 330

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL**
1-Chloronaphthalene	8270(3550)	2	10-150	0-50	330
2-Chloronaphthalene	8270(3550)/CLP-2/88; 3/90	2/6/62	60-118	0-40	330
2-Chiorophenol (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	45-105 25-102	0-31 0-50	330 330
4-Chlorophenylphenyl ether	8270(3550)/CLP-2/88; 3/90	2/6/62	25-158	0-33	330
Chrysene	8270(3550)/CLP-2/88; 3/90	2/6/62	40-148	0-27	330
m-Cresol	8270(3550)	2	10-150	0-50	330
o-Cresol	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330
p-Cresol	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330
4,4'-DDD	8270(3550)	2	10-145	0-40	330
4,4'-DDE	8270(3550)	2	10-136	0-40	330
4,4'-DDT	8270(3550)	2	10-203	0-62	330
Diallate	8270(3550)	2	10-150	0-50	330
Dibenz(a,h)anthracene	8270(3550)/CLP-2/88; 3/90	2/6/62	40-147	0-28	330
Dibenzofuran	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330
Di-n-butylphthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-118	0-50	330
1,2-Dichlorobenzene	8270(3550)/CLP-2/88; 3/90	2/6/62	32-129	0-40	330
1,3-Dichlorobenzene	8270(3550)/CLP-2/88; 3/90	2/6/62	10-172	0-42	330
1,4-Dichlorobenzene (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	46-112 28-104	0-28 0-27	330 330
3,3'-Dichlorobenzidine	8270(3550)/CLP-2/88 CLP 3/90	2/6 62	10-262	0-100	660 330
2,4-Dichlorophenol	8270(3550)/CLP-2/88; 3/90	2/6/62	39-135	0-40	330
2,6-Dichlorophenol	8270(3550)	2	10-150	0-50	330
Dieldrin	8270(3550)	2	29-136	0-40	330
Diethylphthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-114	0-40	330
p-(Dimethylamino)azobenzene	8270(3550)	2	10-150	0-50	330
7,12- Dimethylbenz(a)anthracene	8270(3550)	2	10-150	0-50	330
3,3'-Dimethylbenzidine	8270(3550)	2	10-200	0-100	1700
a,a-Dimethylphenethylamine	8270(3550)	2	10-150	0-50	1700
2,4-Dimethylphenol	8270(3550)/CLP-2/88; 3/90	2/6/62	15-151	0-22	330
Dimethylphthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-112	0-40	330
m-Dinitrobenzene	8270B(3550)	2	10-150	0-50	330
4,6-Dinitro-2-methylphenol	8270(3550)/CLP-2/88 CLP 3/90	2/6 62	10-181	0-93	1700 800

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL ** (ug/kg)	
2,4-Dinitrophenol	8270(3550)/CLP-2/88 CLP 3/90	2/6 62	10-167	0-87	1700 800	
2,4-Dinitrotoluene (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	35-111 28-89	0-29 0-47	330 330	
2,6-Dinitrotoluene	8270(3550)/CLP-2/88; 3/90	2/6/62	50-158	0-40	330	
Dinoseb (2-sec-Butyl-4,6- dinitrophenol)	8270(3550)	2	10-150	0-50	330	
Di-n-octylphthalate	8270(3550)/CLP-2/88; 3/90	2/6/62	10-146	0-50	330	
1,4-Dioxane	8270***V(3550)	2	10-150	0-50	330	
Diphenylamine/ N-nitrosodiphenylamine	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330	
1,2-Diphenyl hydrazine	8270(3550)	2	10-150	0-50	330	
Endosulfan I	8270(3550)	2	10-150	0-50	660	
Endosulfan Il	8270(3550)	2	10-150	0-50	660	
Endosulfan sulfate	8270(3550)	2	10-107	0-50	660	
Endrin	8270(3550)	2	10-150	0-50	660	
Endrin aldehyde	8270(3550)	2	10-209	0-50	1700	
Endrin ketone	8270/(3550)	2	10-150	0-50	1700	
Ethyl methanesulfonate	8270(3550)	2	10-150	0-50	330	
Fluoranthene	8270(3550)/CLP-2/88; 3/90	2/6/62	54-135	0-21	330	
Fluorene	8270(3550)/CLP-2/88; 3/90	2/6/62	59-121	0-40	330	
Heptachlor	8270(3550)	2	10-192	0-40	660	
Heptachlor epoxide	8270(3550)	2	26-155	0-55	660	
Hexachtorobenzene	8270(3550)/CLP-2/88; 3/90	2/6/62	10-152	0-40	330	
Hexachlorobutadiene	8270(3550)/CLP-2/88; 3/90	2/6/62	24-116	0-40	330	
Hexachlorocyclopentadiene	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330	
Hexachtoroethane	8270(3550)/CLP-2/88; 3/90	2/6/62	40-113	0-40	330	
Hexach Lorophene ¹	8270(3550)	2	10-200	0-80	170,000	
Hexachtoropropene	8270(3550)	2	10-150	0-50	330	
Indeno(1,2,3-cd)pyrene	8270(3550)/CLP-2/88; 3/90	2/6/62	18-157	0-83	330	
Isophorone	8270(3550)/CLP-2/88; 3/90	2/6/62	21-196	0-60	330	
Isosafrole	8270(3550)	2	10-150	0-50	330	
Methapyrilene	8270(3550)	2	10-150	0-50	3300	

Exhibits non-reproducible chromatographic behavior.

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER NETHED REFERENCE ACCURACY* PRECISION*					
PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	(% RPD)	PQL** (ug/kg)
3-Methylcholanthrene	8270(3550)	2	10-150	0-50	330
Methyl methanesulfonate	8270(3550)	2	10-150	0-50	330
1-Methylnaphthalene	82708 (3550)	2	10-150	0-50	330
2-Methylnaphthalene	8270(3550)/CLP-2/88; 3/90	2/6/62	10-150	0-50	330
Naphthalene	8270(3550)/CLP-2/88; 3/90	2/6/62	53-125	0-21	330
1,4-Napthoquinone	8270(3550)	2	10-150	0-50	330
1-Napthylamine	8270(3550)	2	10-150	0-50	330
2-Napthylamine	8270(3550)	2	10-150	0-50	330
Nicotine	8270(3550)	2	10-150	0-50	3300
2-Nitroaniline	8270(3550)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	1700 800
3-Nitroaniline	8270(3550)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	1700 800
4-Nitroaniline	8270(3550)/CLP-2/88 CLP-3/90	2/6 62	10-150	0-50	1700 800
Ni trobenzene	8270(3550)/CLP-2/88; 3/90	2/6/62	35-180	0-40	330
2-Nitrophenol	8270(3550)/CLP-2/88; 3/90	2/6/62	29-182	0-40	330
4-Nitrophenol (MS)	8270(3550) CLP-2/88 CLP-3/90	2 6 62	10-130 11-114 11-114	0-34 0-50 0-50	1700 1700 800
4-Nitroquinoline-1-oxide	8270(3550)	2	10-150	0-50	3300
N-Nitroso-di-n-butylamine	8270(3550)	2	10-150	0-50	330
N-Nitrosodiethylamine	8270(3550)	2	10-150	0-50	330
N-Nitrosodimethylamine	8270(3550)	2	10-150	0-50	330
N-Nitroso-di-n-propylamine (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	27-140 41-126	0-35 0-38	330 330
N-Nitrosomethylethylamine	8270(3550)	2	10-150	0-50	330
N-Nitrosomorpholine	8270(3550)	2	10-150	0-50	330
N-Nitrosopiperidine	8270(3550)	2	10-150	0-50	330
H-Hitrosopyrrolidine	8270(3550)	2	10-150	0-50	330
5-Nitro-o-toluidine	8270(3550)	2	10-150	0-50	330
PC8 1016	8270(3550)	2	10-150	0-50	17000
PCB 1221	8270(3550)	. 2	10-150	0-50	17000
PCB 1232	8270(3550)	2	10-150	0-50	17000
PCB 1242	8270(3550)	2	10-150	0-50	17000
PCB 1248	8270(3550)	2	10-150	0-50	17000

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD	REFERENCE	ACCURACY*	PRECISION*	PQL
PCB 1254	(Prep) 8270(3550)	2	(% Rec)	(% RPD)	(ug/kg)
PCB 1260		2			17000
	8270(3550)		10-150	0-50	17000
Pentachlorobenzene	8270(3550)	2	10-150	0-50	330
Pentach Loron i trobenzene	8270(3550)	2	10-150	0-50	330
Pentachlorophenol (MS)	achlorophenol (MS) 8270(3550) CLP-2/88		10-107 17-109	0-89 0-47	1700 1700
	CLP-3/90	62	17-109	0-47	800
Phenacetin	8270(3550)	2	10-150	0-50	330
Phenanthrene	8270(3550)/CLP-2/88; 3/90	2/6/62	56-129	0-21	330
Phenoi (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	37-112 26-90	0-36 0-35	330 330
p-Phenylenediamine	8270(3550)	2	10-150	0-50	1700
2-Picoline	8270(3550)	2	10-150	0-50	330
Pronamide	8270(3550)	2	10-150	0-50	330
Pyrene (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	33-139 35-142	0-25 0-36	330 330
Pyridine	8270(3550)	2	10-150	0-50	330
Safrole	8270(3550)	2	10-150	0-50	3 3 0
Strychnine	8270(3550)	2	10-150	0-50	3300
Trichtorophenols	8270(3550)	22	NA	NA NA	1700
1,2,4,5-Tetrachlorobenzene	8270(3550)	2	10-150	0-50	330
2,3,4,5-Tetrachlorophenol	8270***(3550)	2	10-150	0-50	1700
2,3,4,6-Tetrachlorophenol	8270(3550)	2	36-121	0-31	1700
o-Toluidine	8270(3550)	2	10-150	0-50	330
Toxaphene	8270(3550)	2	10-150	0-50	67000
1,2,4-Trichlorobenzene (MS)	8270(3550) CLP-2/88; 3/90	2 6/62	48-107 38-107	0-28 0-23	330 330
Tetrachlorophenols	8270(3550)	2	NA	NA	1700
2,4,5-Trichlorophenot	8270(3550)/CLP-2/88 CLP-3/90	2/6 62	39-123	0-27	1700 800
2,4,6-Trichlorophenol	8270(3550)/CLP-2/88; 3/90	2/6/62	37-144	0-40	330
o,o,o- Triethylphosphorothicate	8270(3550)	2	10-150	0-50	330
1,3,5-Trinitrobenzene	8270(3550)	2	10-150	0-50	330
Surrogate - Nitrobenzene-d5	8270(3550) CLP-2/88; 3/90	2 6/62	22-124 23-120	NA NA	NA NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRÉCISION* (% RPD)	PQL** (ug/kg)
Surrogate -	8270(3550)	2	35-116	NA	HA
2-Fluorobiphenyl	CLP-2/88; 3/90	6/62	30-115	NA	NA
Surrogate -	8270(3550)	2	29-137	NA	NA
p-Terphenyl-d14	CLP-2/88; 3/90	6/62	18-137	NA	NA
Surrogate -	8270(3550)	2	32-123	NA	NA
Phenol-d5	CLP-2/88; 3/90	6/62	24-113	NA	NA
Surrogate -	8270(3550)	2	27-120	NA	NA
2-Fluorophenol	CLP-2/88; 3/90	6/62	25-121	NA	NA
Surrogate -	8270(3550)	2	17-123	NA	NA
2,4,6-Tribromophenol	CLP-2/88; 3/90	6/62	19-122	NA	NA
Surrogate - 2-Chlorophenol-d4	CLP-3/90	62	20-130	NA	NA
Surrogate - 1,2-Dichlorobenzene-d4	CLP-3/90	62	20-130	NA	AK

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p- dioxin (2,3,7,8-TCDD)	8280 8270 (Qual. Screen)	2 2	69-145	0-40	0.50 330
Polychlorinated Dibenzo-p-dioxin	s and Dibenzofurans clas	ses			
tetra-CDD	8280	2	69-145	0-40	0.50
tetra-CDF	8280	2	59-142	0-40	0.50
penta-CDD	8280	2	41-203	0-40	0.50
penta-CDF	8280	2	55-146	0-40	0.50
hexa-CDD	8280	2	45-174	0-53	0.50
hexa-CDF	8280	2	50-154	0-46	0.50
hepta-CDD	8280	2	20-170	0-50	1.0
hepta-CDF	8280	2	20-170	0-50	1.0
octa-CDD	8280	2	20-170	0-50	1.0
octa-CDF	8280	2	20-170	0-50	1.0
Internal Standard - "2C,,-2,3,7,8-TCDD	8280	2	40-120	NA .	NA
Internal Standard - ""C ₁₂ -OCDD	8280	2	40-120	NA I	NA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	NETHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Aminocarb	632(3550)***	13/2	50-150	0-50	20
Barban	632(3550)***V	13/2	50-150	0-50	50
Bromacit	632(3550)***	13/2	50-150	0-50	40
Carbaryl (MS)	632(3550)***V	13/2	50-150	0-50	50
Carbofuran	632(3550)***V	13/2	50-150	0-50	50
Chlorpropham	632(3550)***V	13/2	50-150	0-50	20
Diuron (MS)	632(3550)***V	13/2	50-150	0-50	5.0
Fenuron	632(3550)***V	13/2	50-150	0-50	10
Fluometuron	632(3550)***V	13/2	50-150	0-50	10
Linuron	632(3550)***V	13/2	50-150	0-50	5.0
Hethiocarb	632(3550)***V	13/2	50-150	0-50	50
Methomyl	632(3550)***V	13/2	50-150	0-50	200
Honuron	. 632(3550)***V	13/2	50-150	0-50	5.0
Neburon	632(3550)***V	13/2	50-150	0-50	5.0
Oxamyl	632(3550)***V	13/2	50-150	0-50	50
Propachlor	632(3550)***	13/2	25-148	NA NA	NA
Propham	632(3550)***V	13/2	50-150	0-50	50
Ргорохиг	632(3550)***V	13/2	50-150	0-50	50
Siduron	632(3550)***V	13/2	50-150	0-50	20
Sнер	632(3550)***	13/2	50-150	0-50	20

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

	<u> </u>		<u> </u>		
PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQ[** (ug/kg)
2,4-0	644(3550)***	64/2	40-150	0-50	67
2,4-DB	644(3550)***	64/2	40-150	0-50	33
Dicamba	644(3550)***	64/2	40-150	0-50	17
Picloram	644(3550)***	64/2	40-150	0-50	17

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acenaphthene (MS)	8310	2	11-144	0-35	20
Acenaphthylene	8310	2	10-139	0-40	20
Acridine	8310***	2	10-200	0-40	10
Anthracene	8310	2	10-126	0-40	4.0
Benzo(a)anthracene	8310	2	12-135	0-40	4.0
Benzo(b)fluoranthene	8310	2	10-150	0-40	4.0
Benzo(k)fluoranthene	8310	2	10-159	0-40	10
Benzonitrile	8310***	2	10-200	0-40	200
Benzo(g,h,i)perylene	8310	2	10-120	0-40	10
Benzo(a)pyrene	8310	2	10-128	0-40	4.0
7,8-Benzoquinoline	8310***	2	10-200	0-40	20
Carbazole	8310***	2	10-150	0-40	20
Chrysene (MS)	8310	2	10-199	0-40	4.0
Dibenzo(a,h)anthracene	8310	2	10-110	0-40	20
2,4-Dimethylauinoline	8310***	2	10-200	0-40	400
fluoranthene	8310	2	56-136	0-28	10
Fluorene (MS)	8310	2	10-142	0-40	10
Indeno(1,2,3-cd)pyrene	8310	2	10-116	0-40	10
1-Methylnaphthalene	8310	2	10-125	0-40	20
2-Methylnaphthalene	8310	2	10-125	0-40	20
8-Methylquinoline	8310***	2	10-200	0-40	100
Naphthalene (MS)	8310	2	31-159	0-34	20
Phenanthrene	8310	2	10-155	0-40	4.0
Pyrene (MS)	8310	2	49-156	0-28	10
Quinaldine	8310***	2	10-200	0-40	100
Quinoline	8310***	2	10-200	0-40	800
Surrogate - 2-Fluorobiphenyl	8310	2	60-140	AK	NA.
Surrogate - 4-Terphenyl-d4	8310	2	60-140	на	XA

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (ug/kg)
Acetaldehyde	8315	2	30-110	0-40	2000
Formaldehyde	8315	2	50-155	0-40	100
Aldicarb (Temik) (MS)	8318	2	44-114	0-50	20
Aldicarb sulfone	8318	2	58-118	0-50	50
Aldicarb sulfoxide	8318***	2	33-143	0-50	50
Carbofuran (Furadan) (MS)	8318	_2	53-123	0-50	30
Carbaryl (Sevin)	8318	2	56-126	0-50	50
Dioxacarb	8318	2	55-125_	0-50	100
Ethylene thiourea	8318***	2	30-140	0-50	330
3-Hydroxycarbofuran	8318	2	60-120	0-50	20
Methiocarb (Mesuroi)	8318	2	52-122	0-50	50
Methomyl (Lannate)	8318	2	54-114	0-50	20
Oxamyl (MS)	8318***	2	45-161	0-50	50
Promecarb	8318	2	44-120	0-50	20
Propoxur (Baygon)	8318	2	46-116	0-50	20

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TABLE 5.2. LABORATORY ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR SOLIDS AND SEMISOLIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (Ug/L)
1,3-Dinitrobenzene (MS)	8330	2	54-166	0-30	100
2,4-Dinitrotoluene (MS)	8330	2	60-140	0-30	100
2,6-Dinitrotoluene	8330	2	60-140	0-30	100
Diphenylamine	8330***v	2	65-140	0-30	100
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	8330	2	54-166	0-30	50
Methyl-2,4,6-trinitro-phenylnitramine (Tetryl)	8330	2	41-165	0-30	200
Nitrobenzene	8330	2	52-152	0-30	50
Nitroglycerin	8330***V	2	46-190	0-50	1000
n-Nitrosodiphenylamine	8330***v	2	55-121	0-30	100
2-Nitrotoluene (MS)	8330	2	50-144	0-30	200
3-Nitrotoluene	8330	2	55 - 165	0-30	100
4-Witrotoluene	8330	2	54-166	0-30	200
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	8330	2	54-162	0-30	500
1,3,5-Trinitrobenzene	8330	2	50-150	0-30	100
2,4,6-Yrinitrotoluene	8330	2	50-170	0-30	200
Surrogate - 2-fluorobiphenyl	8330	2	40-140	NA	NA

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TABLE 5-3. FIELD ANALYTICAL METHODS, QA OBJECTIVES AND PRACTICAL QUANTITATION LIMITS (PQL) FOR WATER AND OTHER LIQUIDS

PARAMETER	METHOD (Prep)	REFERENCE	ACCURACY* (% Rec)	PRECISION* (% RPD)	PQL** (mg/L)
Chlorine, residual	330.5	3	NA NA	0-40	1.0
Hydrogen ion (pH)	150.1/9040	3/2	85-115	0-15	NA NA
Oxygen (dissolved)	360.1	3	, NA	0-30	0.20
Salinity	210	4	NA.	NA	100
Specific conductance	120.1/9050	3/2	90-110	0-10	5.0 umho/cm
Temperature	170.1	3	NA	0-10	NA NA
Turbidity	180.1/214A	3/4	60-140	0-30	0.10 NTU
Water level	EPA	12	NA NA	0-5	0.10 ft.

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REFERENCES AND NOTES FOR TABLES 5.1, 5.2, AND 5.3

Accuracy and precision control limits are primarily derived from inhouse laboratory data. For inorganic parameters, accuracy and precision control limits that have been generated from historical data have been rounded to the nearest "5". In some cases, published limits may be used in lieu of in-house limits because insufficient in-house data are available to calculate limits. In cases where insufficient data are available to generate in-house limits, and no EPA-approved method limits exist, limits are estimated based on available data. In-house data will be generated for all parameters by the next annual revision of this plan.

- ** PQL Practical Quantitation Limits These are the normal reporting limits for routine environmental samples. In all cases, PQLs are higher than laboratory established Method Detection Limits (MDL). These PQLs are taken from SW-846 (Third Edition) or derived from inhouse data on routine environmental samples. If samples are highly contaminated or contain interfering substances, PQLs may be elevated by a dilution factor.
- *** This compound is not included in EPA's list of compounds for this method. However, Savannah Laboratories has verified (validated) that this coupound can be analyzed by this method and will report data for this compound if specifically requested by the client.
- ***V Method validation data for this compound are included in Appendix A.
- 1. Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office: Washington, DC, July 1, 1988.
- Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November, 1986.
- Methods for Chemical Analysis of Water and Wastes; U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983; EPA 600/4-79-020.
- 4. Standard Methods for the Examination of Water and Wastewater, Sixteenth and Seventeenth Editions; American Public Health Association: Washington, DC, 1985 and 1989.
- Deepwater Ports Maintenance, Dredging, and Disposal Manual; Florida DER.

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- 6. CLP US EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Medium, Multi-Concentration, Revision 2/88.
- 7. Determination of Triazine Pesticides in Industrial and Municipal Wastewater: EPA Method 619; January, 1982.
- 8. Determination of Thiophosphate Pesticides in Industrial and Municipal Wastewater: EPA Method 622.1; January, 1982.
- 9. Determination of Dinitroaniline Pesticides in Industrial and Municipal Wastewater: EPA Method 627; January, 1982.
- 10. Determination of Organochlorine Pesticides in Industrial and Municipal Wastewater: EPA Method 608.1; February, 1982.
- 12. Analytical Procedures for Detection and Quantification of Total Petroleum Fuel Hydrocarbons and Fuel Constituents: Calif. Method for Modified 8015; Don M. Eisenberg, Adam W. Olivier, Peter W. Johnson, Daniel S. Tempelis; September, 1985.
- 13. Determination of Carbamate and Urea Pesticides in Industrial and Municipal Wastewater: EPA Method 632; January, 1982.
- 14. Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: EPA Method 622; January, 1982.
- 15. Determination of Thiocarbamate Pesticides in Industrial and Municipal Wastewaters by Gas Chromatography: EPA Method 634; January, 1982.
- 16. Determination of Bensulide in Industrial and Municipal Wastewaters by Liquid Chromatography: EPA Method 636.
- 17. Determination of Mercaptobenzothiazole in Wastewaters by Liquid Chromatography: EPA Method 640.
- 18. Determination of Hexachlorophene and Dichlorophen in Industrial and Municipal Wastewaters: EPA Method 604.1.
- 19. Determination of Rotenone in Industrial and Municipal Wastewaters by Liquid Chromatography: EPA Method 635.
- 20. Determination of Bendiocarb in Industrial and Municipal Wastewaters by Liquid Chromatography: EPA Method 639.
- Determination of Oryzalin in Industrial and Municipal Wastewaters: EPA Method 638.
- 22. Determination of MBTS and TCMTB in Industrial and Municipal Wastewater by Liquid Chromatography: EPA Method 637.
- 23. Determination of Diphenylamine in Industrial and Municipal Wastewater by Gas Chromatography: EPA Method 620.
- 24. C, H, and O Compounds: EPA Method 616.

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- 25. Determination of Cyanazine in Industrial and Municipal Wastewater: EPA Method 629; January, 1982.
- 26. Determination of Organohalide Pesticides and PCBs in Industrial and Municipal Wastewater: EPA Method 617; January, 1982.
- 27. Determination of Volatile Pesticides in Municipal and Industrial Wastewater by Gas Chromatography: EPA Method 618.
- 28. Analysis of Certain Amine Pesticides and Lethane in Wastewater by Gas Chromatography: EPA Method 645.
- 30. Measurement of Trihalomethanes in Drinking Water with Gas Chromatography/ Mass Spectrometry and Selected Ion Monitoring: EPA Method 501.3.
- 31. Method from FDER Central Lab
- 33. Measurement of N-Methyl Carbamoyloximes and N-Methyl Carbamates in Drinking Water by Direct Aqueous Injection HPLC with Post Column Derivatization: EPA Method 531.
- 35. NIOSH National Institute for Occupational Safety and Health, Third Edition, 1987.
- 36. Official Methods of Analysis of the Association of Official Analytical Chemists, Method for Formaldehyde 20.063 (Chromotropic Acid), Thirteenth Edition, 1980.
- 38. Annual Book of ASTM Standards, Part 23; ASTM: Philadelphia, PA, 1980.
- Annual Book of ASTM Standards, Volume 11.01/11.02; ASTM: Philadelphia, PA, 1989.
- 40. Balls, P.W.; Atomic Absorption Spectrometric/Hydride Generation Determination of Tributyl Tin and Dibutly Tin in Sea Water at the Nanogram per Liter Level; ANALYTICA CHEMICA ACTA 197; 309-313 (1987).
- 41. Determination of Organophosphorus Pesticides in Industrial and Municipal Wastewater: EPA Method 633.
- 42. Methods for the Determination of Organic Substances in Water and Fluvial Sediments, USGS Book 5, 1983.
- 43. Methods of Soil Analysis, American Society of Agronomy, Inc., Number 9, Part 2, page 570, (Walkley-Black Procedure).
- 44. EPA 600/4-84-008, Appendix D: Method For Extractable Organic Halides (EOX) In Solids, January, 1984.
- 45. CLP-US EPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi-concentration; Revisions 7/88 and 3/90.
- 46. EPA/CE-81-1 Technical Report, May 1981: Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material; Procedures for Handling and Chemical Analysis of Sediment and Water Samples.

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- 48. Code of Federal Regulations, Title 40, Part 268; U.S. Government Printing Office: Washington, DC, November 7, 1986.
- 49. Analysis of THMs in Finished Waters by the Purge and Trap Method: EPA Method 501.1.
- 50. Analysis of THMs in Drinking Water by Liquid/Liquid Extraction; EPA Method 501.2.
- 51. EPA 600/4-88-039: Methods for the Determination of Organic Compounds in Drinking Water, December, 1988.
- 52. Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater: EPA Method 614, February, 1982.
- 53. Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater: EPA Method 615, 1982.
- 54. EPA 600/4-80-032: Prescribed Procedures for Measurement of Radioactivity in Drinking Water, August, 1980.
- 55. Determination of Benomyl and Carabendazim in Wastewater: EPA Method 631.
- 56. Simon, Verne A.; A Novel Method for the Determination of Paraquat and Diquat in Water by HPLC; Florida HRS.
- 57. Pesticides in Wastewater: EPA Method 608.2.
- 58. Determination of Organophosphorus Pesticides in Wastewater: EPA Method 614.1.
- 59. Analysis of Bentazon in Wastewater by Liquid Chromatography: EPA Method 643.
- 60. Calculation of Un-Ionized Ammonia in Fresh Water; Florida DER, October, 1983.
- 61. Bellar, T.A., and Lichtenberg, J. J.; The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils; U.S. EPA Environmental Monitoring and Support Laboratory: Cincinnati, OH, September, 1982; EPA-600/4-81-045.
- 62. CLP US EPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, Revision OLM01.0 (3/90) thru OLM01.6 (6/91).
- 63. Determination of Dithiocarbamate Pesticides in Industrial and Municipal Wastewater: EPA Method 630.
- 64. Analysis of Picloram in Wastewater by Liquid Chromatography; EPA Method 644.
- 65. Inductively Coupled Plasma Atomic Emission Analysis of Drinking Water, Appendix to Method 200.7, Revision 1.3; USEPA, March, 1987.

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6.0 SAMPLING PROCEDURES

When Savannah Laboratories has field sampling responsibilities, an experienced field sampling crew will be sent to the site for sample collection and delivery of samples to the laboratory. Each crew will be supervised by a highly qualified field sampling manager who is trained according to EPA and DER protocol for groundwater and other environmental sampling. On past projects, these managers have had their field sampling techniques critiqued by FDER personnel (Bureau of Groundwater Protection), Georgia EPD personnel, and EPA Region IV field coordinators.

The DER Interim Field Activity SOPs have been adopted by Savannah Laboratories. The notarized statement of intent is found at the end of this section.

6.1 Sampling Capabilities

Savannah Laboratories has the capability for sampling groundwater, surface water, wastewater, soils, sediments/sludges, drinking water, and tissues for the following analyte classes:

Analyte Class	Sample Source
Volatile Organics (VCCs)	Drinking water, groundwater, surface water, wastewater, soils, sediment, and tissues
Semivolatile Organics	Drinking water, groundwater, surface water, wastewater, soils, sediment, and tissues
Pesticides/Herbicides/PCBs	Drinking water, groundwater, surface water, wastewater, soils, sediment, and tissues
Metals (total and/or dissolved)	Drinking water, groundwater, surface water, wastewater, soils, sediment, and tissues
Coliform (total/fecal)	Drinking water, groundwater, surface water, wastewater, soils, sediment and tissues
Cyanide/Sulfide	Drinking water, groundwater, surface water, wastewater, soils and sediment
TRPH, TPH(1)	Drinking water, groundwater, surface water, wastewater, soils and sediment
Nutrients(2)	Drinking water, groundwater, surface water, wastewater, soils and sediment
General: pH, specific conductance, temperature, turbidity, TSS, TDS, TOC, DO, COD, BOD	Drinking water, groundwater, surface water, wastewater, soils and sediment
Footnotes: (1) TRPH = Total Recoverable Petroleu TPH = Total Petroleum Hydrocarbo	

(2) Nutrients = Nitrogen, Phosphorus Series; Chloride, Sulfate

6.2 Sampling Equipment

Sampling equipment conforms to construction and usage conditions detailed in the DER General Sampling Protocols SOP, Revised October 29, 1991. A specific equipment listing is provided at the beginning of each subsection of "Sampling Procedures" (Section 6.4).

Following is a list of other routinely used equipment.

Item	Use
Ice chests, styrofoam or insulated plastic	Sample container and sample transport
Sampling vehicles	Sample container and sample transport
Field thermometer	Field measurement of temperature
Field pH meter	Field measurement of pd
Field conductivity meter	Field measurement of conductivity
Electronic water level indicator	Well volume calculation
Stainless steel tape measure	Well volume calculation
Nylon line	Well volume calculation
Sheet plastic	Contamination control
Aluminum foil	Contamination control
Plastic or metal buckets	Collection of purge water or cleaning wastes
Cleaning brushes	Equipment decontamination
Liquinox detergent	Equipment decontamination
Analyte-free water contained in contaminant-free glass or plastic bottles	Equipment decontamination
Isopropyl alcohol (nanograde) contained in contaminant- free glass or plastic bottles	Equipment decontamination
10% Nitric acid (metals grade) contained in contaminant-free glass bottles	Equipment decontamination (except for stainless steel equipment)
Glass or plastic jugs	Transport of cleaning wastes
Sample preservation reagents contained in dispenser bottles or reagent bottles	Sample preservation
Field carrier (covered, divided tray or box)	Transport of preservation reagents
pH paper	Field-check of sample preservation
Disposable pipettes	Addition of preservation reagents
Standard buffer solutions	Calibration of field pH meter
Standard KCl solution	Calibration check of field conductivity meter
Disposable unpowdered latex gloves	Contamination control

6.3 Decontamination and Cleaning Procedures

Sample containers will be obtained or cleaned by option 2a or 2b in the DER SOP for Cleaning and Decontaminating Sampling Equipment, revised October 29, 1991.

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Sampling equipment will be cleaned and decontaminated according to protocols outlined in the DER SOP for Cleaning and Decontaminating Sampling Equipment, revised October 29, 1991.

6.4 Sampling Protocols

6.4.1 General Considerations

All sampling will be performed according to the general protocols outlined in the DER General Sampling Protocols SOP, Section I, revised October 29, 1991.

6.4.2 Wastewater Sampling

Wastewater samples will be collected according to the DER SOPs Sampling Procedures for Wastewater and Surface Water, revised October 25, 1991 and General Sampling Protocols, Section III.F, revised October 29, 1991.

Below is a list of equipment available for wastewater sampling and the parameters which may be sampled.

Type	Construction Materials	Use	Permissible Parameters
Autosampler	Silicon tubing, plastic collection vessel	Composite samples	Metals, non-metallic inorganics, nutrients, demands, radiological
	Teflon tubing, glass collection vessel	Composite samples	Organics, non-metallic inorganics, nutrients, demands, radiological
Kenmerer	SS or glass, acrylic stopper	Grab @ specific depth	All inorganics
Bucket, beaker, unpreserved sample bottle, dipper ²	SS. glass or Teflon Plastic	Discrete grab Discrete grab	All inorganics

Three automatic samplers are available among the five divisions. Refrigeration capability is available.

6.4.3 Surface Water Sampling

Surface water samples will be collected according to the DER SOPs Sampling Procedures for Wastewater and Surface Water, revised October 25, 1991 and General Sampling Protocols, Section III.A revised October 29, 1991 and the EPA Region IV Standard Operating Procedures and Quality Assurance Manual, Section 4.8.3, revised February 1991. Below is a list of equipment available for surface water sampling and the parameters which may be sampled.

Device is lowered into stream via decontaminated lines or rods.

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Туре	Material	Use	Permissible Parameters
DO Dunker	SS or glass	discrete grab, depth composite	All
Kemmerer	SS or glass acrylic stopper	grab @ specific depth	Inorganics
Beaker	SS or glass	discrete grab	A11 ·
Bailer	SS or Teflon	grab @ specific depth2	A11
Peristaltic pump with weighted tubing	SS or Teflon silicon tubing	grab at specific depth	Inorganics

Footnotes:

- 1 Beaker is inverted, submerged, then turned over to fill.
- 2 Depth limited by length of bailer.

6.4.4 Groundwater Sampling

Groundwater samples will be collected according to the DER SOP Groundwater Sampling Procedures, revised October 28, 1991.

Below is a listing of pump types and tubing materials used by Savannah Laboratories. Equipment may be interchanged among the five laboratory locations according to need.

Ришр Туре	Units	lise	Parameters	Description
Positive displacement	nt			
Submersible	4	Purging	A11	1
Bladder	2	Purging, sampling	Inorganics	2
Suction lift				
Centrifugal	4	Purging	A11	3
Peristaltic	4	Field filtration, purging	Metals	4

- Submersible pump housing, internal surfaces, and upper fitting for tubing are stainless steel. A 4' to 8' length of Teflon tubing is attached to the stainless steel fitting. The remainder of the discharge tubing is garden hose. The suspension cable is 3' to 4' of stainless steel or Teflon-coated stainless steel, attached to a nylon rope. A check valve at the upper stainless steel/Teflon junction prevents backflow of purge water into the well.
- 2. The bladder pump housing is Lexan plastic and the tubing is polyethylene. This pump is used for purging only in the case of 2" diameter deep wells. After bladder pump purging, one well volume is purged with an appropriate bailer prior to sampling.
- Centrifugal surface pumps utilize 4' joinable sections of PVC pipe with a 3' to 4' Teflon tail piece. Only the Teflon portion contacts

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the formation water. A foot valve prevents backflow of purge water into the well.

4. Peristaltic pumps are routinely used only for in-line field filtration of metals samples. Tubing may be medical grade silicone, Tygon, or polypropylene flexible tubing. On rare occasions, a small diameter shallow well may be purged using this pump. In this case, a Teflon tailpipe arrangement would be used, with only the Teflon contacting the formation water. To prevent backflow of purge water, the tubing is withdrawn from the well while the pump is running.

Below is a listing of bailer materials available for groundwater sampling.

Bailer Material	Permissible Parameters	Non-permissible Parameters
PVC	Metals; non-metallic inorganics: nutrients, demands; biological	Organics, volatile or extractable
Stainless Steel	All parameters	None
Teflon	All parameters	None
Clear PVC or acrylic	Free product thickness	

6.4.5 Potable Water Sampling

Potable water samples will be collected according to the DER SOP Groundwater Sampling Procedures, revised October 28, 1991, and the EPA Region IV SOP and QAM, Section 4.10.2, revised February 1991. Equipment available for potable water sampling is listed under groundwater sampling (6.4.4).

6.4.6 Sampling for Soil and Sediment

Soil samples will be collected according to the DER SOPs Soil Sampling Procedures, Revised October 28, 1991, and General Sampling Protocols, Section III.D, revised October 28, 1991.

Sediments will be collected according to the DER SOP General Sampling Protocols, Section III.B, revised October 29, 1991 and the EPA Region IV SOP and QAM, Section 4.8.3.3, revised February 1991.

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Below is a list of soil and sediment sampling devices used by Savannah Laboratories.

Туре	Material	Use	Permissible Parameters
Trowel, spoon	SS Teflon-coated SS	sampling	All
Shovel	Aluminum SS	sampling sampling	Demands, nutrients Metals, organics
Corer	SS PVC pipe	sampling sampling	All Inorganics
Hand auger	SS	sampling	All
Ponar grab sampler	SS	sediment sampling	All
Mixing tray	Metal, foil-lined glass Plastic	homogenizing, compositing homogenizing compositing	Extractable organics Inorganics

6.4.7 Sludge Sampling

Domestic waste residual sludges will be collected according to the EPA POTW Sludge Sampling and Analysis Guidance Document, revised August 1989.

Sludges from solid and hazardous waste sites will be collected according to the *EPA Region IV SOP and QAM*, Sections 4.12.3 and 4.12.5, revised February 1991.

6.4.8 Liquid Hazardous Waste

Hazardous wastes, drums, and tanks of unknown origins and concentrations are typically not sampled by Savannah Laboratories because the sample operations are inherently dangerous to the personnel involved. Drums and tanks are occasionally sampled when the primary constituents are known and do not present a toxic, fire, or explosion hazard.

If drum tank or pit sampling is undertaken, it is performed according to the *EPA Region IV SOP and QAM*, Sections 4.12.3 and 4.12.4, Revised February 1991.

6.4.9 Biological Specimens and Tissues

Fish tissues are prepared for analysis according to DER QA Guidance Document #90-01, revised August 15, 1990, using properly decontaminated stainless steel implements.

Other biological specimens are obtained and prepared in a manner which will preclude contamination from implements or other specimens.

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6.5 Special Sampling Considerations

Details of sampling such as compositing, duplicate or split samples, filtration, and special procedures for volatiles, oil and grease, and microbiological samples will be observed as outlined in the DER SOP General Sampling Prococols, Section IV.A through .E, revised October 29, 1991.

6.6 Sample Preservation and Holding Times

Sample preservation, holding times, required sample volumes, and container types are listed in Table 6.1 for water samples and Table 6.2 for soil and sediment samples. These tables are taken from 40 CFR Part 136, Table II for water, and DER QAS Guidance Document # 90-02 for soil. Table 6.3 lists the approved procedures, preservation, and holding times for water for parameters not listed on Table 6.1.

6.7 Sample Preservation Protocols

Sample preservation will be accomplished by option V.A.l of the DER SOP General Sampling Protocols, revised October 29, 1991. The efficacy of the preservation will be checked at the laboratory immediately upon receipt, for all preserved samples except volatiles. Necessary adjustments will be made and recorded in a laboratory logbook. The pH of volatiles samples will be checked upon analysis, unless the client requests a sacrificial vial to be checked upon receipt.

Special preservation protocols will be followed as outlined in the DER SOP General Sampling Protocols, Section V.B.2.a, b, and c, revised October 29, 1991.

6.8 Sample Dispatch and Recordkeeping

Samples will be labeled, packed, and shipped according to the DER SOP General Sampling Protocols, Section V.C. revised October 29, 1991. Examples of a sample label, monitoring well sampling log, and chain-of-custody forms are present in Figures 6.1, 6.2, and 6.3.

See Section 7 for sample custody procedures.

6.9 Field Reagent and Standard Storage

All reagents, standards, and solvents used in field activities are stored and transported as listed in Table 6.4 and according to the DER SOP General Sampling Protocols, Section VI.A, revised October 29, 1991.

TABLE 6.1

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WATER SAMPLES

PARAMETER	SAMPLE CONTAINER ¹	SAMPLE PRESERVATION ^{2,3}	RECOMMENDED HOLDING TIMES
Bacterial Tests:			
Coliform, fecal and total	250-mL P	Cool, 4°C, 0.0081 Na2S203	6 hours
Fecal streptococci	250-mL P	Cool, 4°C, 0.008% Na,S,O,3	6 hours
Inorganic Tests:			
Acidity	250-mL P	Cool, 4°C	14 days
Alkalinity	250-mL P	Cool, 4°C	14 days
Ammonia	100-mL P	Cool, 4°C, H,SO, to pH < 2	28 days
Biochemical oxygen demand	1-L P	Cool, 4°C	48 hours
Bromide	100-mL P	None required	28 days
Biochemical oxygen demand, carbonaceous	1-L P	Cool, 4°C	48 hours
Chemical oxygen demand	100-mi P	Cool, 4°C, H2SO, to pH < 2	28 days
Chloride	100-mL P	None required	28 days
Chlorine, total residual	250-mL amber G	None required	Analyze immediately
Color	250-mL P	Cool, 4°C	48 hours
Cyanide, total and amenable to chlorination	1-L P	Cool, 4°C, NaOH to pH > 12, 0.6 g ascorbic acid	14 days*
Fluoride	100-mL P	None required	28 days
Hardness	250-mL P	HNO, to pH < 2, H ₄ SO, to pH < 2	6 months
Hydrogen ion (pH)	100-mL P	None required	Analyze immediately
Kjeldahl and organic nitrogen	250-mL P	Cool, 4°C, H ₂ SO, to pH < 2	28 days
Chromium VI	250-mL P	Cool, 4°C	24 hours
Mercury'	130-mL G	ENO, to pH < 2	28 days
Metals', except chromium VI and mercury	250-mL P	ENO, to pE < 2	6 months
Nitrate	100-mL P	Cool, 4°C	48 hours
Nitrate-nitrite	100-mL P	Cool, 4°C, H,SO, to pH < 2	28 days
Nitrite	100-mL P	Cool, 4°C	48 hours
Organic carbon	125-mL amber G	Cool, 4°C, HCl or H,SO, to pH < 2	28 days

TABLE 6.1

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WATER SAMPLES

PARAMETER	SAMPLE CONTAINER ¹	SAMPLE PRESERVATION ^{2.3}	RECOMMENDED HOLDING TIMES
Orthophosphate	100-mL P	Filter immediately, cool, 4°C	48 hours
Oxygen, dissolved (Probe)	G bottle & top	None required	Analyze immediately
Winkler	G bottle & top	Fix on site and store in dark	8 hours
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total	250-ml P	Cool, 4°C, H,SO, to pH < 2	28 days
Residue, total	500-mL P	Cool, 4°C	7 days
Residue, filterable (TDS)	500-mL P	Cool, 4°C	7 days
Residue, nonfilterable (TSS)	500-ml P	Cool, 4°C	7 days
Residue, settleable	500-mL P	Cool, 4°C	48-hours
Residue, volatile (VSS)	500~mL P	Cool, 4°C	7 days
Silica	250-mL P	Cool, 4°C	28 days
Specific Conductance	100-mL P	Cool, 4°C	28 days
Sulfate	100-mL P	Cool, 4°C	28 days
Sulfide	250-mL P	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9	7 days
Sulfite	100-ml P	None required	Analyze immediately
Surfactants	1-L P	Cool, 4°C	48 hours
Temperature	100-mL P	None required	Analyze immediately
Turbidity	250-mL P	Cool, 4°C	48 hours
Organic Tests:			
Purgeable halocarbons	4 X 40-mL G, Teflon-lined septum	Cool, 4°C, 0.0081 Na,S,O,° or 0.061 ascorbic acid°	14 days
Purgeable aromatic hydrocarbons	4 X 40-mL G, Teflon-lined septum	Cool, 4°C, 0.0081 Na ₂ S ₂ O ₃ *, HCl to pH < 2° or 0.061 ascorbic acid*	14 days
Acrolein and acrylonitrile	1-L G, Teflon- lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ *, adjust pH to 4-51° or 0.06% ascorbic acid*	14 days
Phenols ¹¹	1-L G, Teflon- lined cap	Cool, 4°C, 0.0081 Na ₂ S ₂ O ₃ 3	Extraction-7 days Analysis-40 days

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TABLE 6.1

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WATER SAMPLES

PARAMETER	SAMPLE CONTAINER ¹	SAMPLE PRESERVATION ^{2,3}	RECOMMENDED HOLDING TIMES*
Benzidines''.12	1-L G, Teflon- lined cap	Cool, 4°C, 0.0081 Na ₂ S ₂ O ₃ 5	Extraction-7 days ¹³
Phthalate esters"	1-L G, Teflon- lined cap	Cool, 4°C	Extraction-7 days Analysis-40 days
Nitrosamines:1:-14	1-L G, Teflon- lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ 5	Extraction-7 days Analysis-40 days
Pesticides"	1-L G, Teflon- lined cap	Cool, 4°C, pH 5-915	Extraction-7 days Analysis-40 days
PCBs ¹¹	1-L G, Teflon- lined cap	Cool, 4°C	Extraction-7 days Analysis-40 days
Nitroaromatics and isophorone"	1-L G, Teflon- lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ 5	Extraction-7 days Analysis-40 days
Polynuclear aromatic hydrocarbons"	1-L G, Teflon- lined cap	Cool, 4°C, 0.008% Na ₃ S ₂ O ₃ S	Extraction-7 days Analysis-40 days
Haloethers'	1-L G, Teflon- lined cap	Cool, 4°C, 0.0081 Na,S ₂ O ₃ 1	Extraction-7 days Analysis-40 days
Chlorinated hydrocarbons11	1-L G, Teflon- lined cap	Cool, 4°C	Extraction-7 days Analysis-40 days
TCDD11	1-L G, Teflon- lined cap	Cool, 4°C, 0.0081 Na,5,0,5	Extraction-30 days Analysis-45 days of collection
Total organic halogens	500-mL amber G, Teflon-lined cap	Cool, 4°C E ₂ SO ₄ to pH < 2	28 days
Total petroleum hydrocarbons	1-L G, Teflon- lined cap	Cool, 4°C EC1 to < 2	28 days
Phenols, total recoverable	1-L G	Cool, 4°C, H,SO, to pR < 2	28 days
Oil and grease	1-L G	Cool, 4°C, R ₂ SO, to pH < 2	28 days
Radiological Tests:			
Alpha, beta and radium	P,G	ENO, to pH < 2	6 Months

- 1. Polyethylene (P) or Glass (G). In cases where more than one inorganic parameter with the sample preservative is required, a single sample container of sufficient size for all analyses is usually preferred. Such grouping of parameters will be indicated when bottles are provided for client sampling.
- 2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, chemical samples may be preserved by maintaining at 4°C until

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compositing and sample splitting are completed.

- 3. When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following: Hydrochloric Acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- 4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5. Sodium thiosulfate or ascorbic acid may be used only if residual chlorine is present. The dechlorination agent and hydrochloric acid must not be combined in pre-preserved vials.
- 6. Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- 7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- 8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- 9. Sample receiving no pH adjustments must be analyzed within seven days of sampling.
- 10. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
- 11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in Footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine), and Footnotes 12 and 13 (re: the analysis of benzidine).

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- 12. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
- 13. Extracts may be stored up to seven days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- 14. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 within 24 hours of sampling.
- 15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

TABLE 6.2

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR SOIL OR SEDIMENT SAMPLES

PARAMETER SAMPLE CONTAINER		SAMPLE PRESERVATION	RECOMMENDED HOLDING TIMES	
Cyanide	500-mi P	Cool to 4°C	14 days	
Sulfide	500- <u>mi</u> P	Cool to 4°C	7 days	
Oil & grease, Total petroleum hydrocarbons	500-mL G	Cool to 4°C	28 days	
Nutrients/TOC	500-mL P	Cool to 4°C	28 days	
Metals (except Mercury)	500-mi P	None required	6 months	
Semivolatile organics, pesticides, etc.	500-mL G with Teflon- lined lid	Dark, cool to 4°C	Extraction-14 days Analysis-within 40 days of extraction	
Volatile organics	125-mi amber G with Teflon-lined lid	Dark, cool to 4°C	14 days	
Mercury	500-mi ?	Cool to 4°C	28 days	

**TABLE 6.3

APPROVED WATER AND WASTEWATER PROCEDURES, CONTAINERS, PRESERVATION AND HOLDING TIMES FOR PARAMETERS NOT FOUND IN 40 CFR 136

Parameter	Method	Reference ¹	Container ²	Preservation ³	Maximum Holding Times ⁴
Bromine	DPD Colorimetric ⁵	SM 408E	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA-SOP (300.0) ⁶	P, G	Cool, 4° C	30 days
Chlorophylls	Spectrophotometric	SM 1002G	P, G ⁷	14 d in dark	30 days'
Corrosivity	Calculated (CaCO ₃ Stability, Langelier Index)	SM 203 ASTM 0513-82	P, G	Cool, 4° C ⁸	7 days ⁸
Odor	Human Panel	SM 207	G only	Cool, 4° C	6 hours
Salinity	Electrometric ⁹ Hydrometric Argentometric	SM 210A	G, wax seal	Analyze immediately or use wax seal	30 days ⁹
Taste	Human Panel	SM 211 A,B ASTM 1292-86	G only	Cool, 4° C	24 hours
Transparency	Irradiometric ¹⁰	17-3.021 FAC			Analyze in-situ
Un-ionized Ammonia	Calculated ¹¹	DER - SOP ¹²	P, G	Cool, 4° C Na ₂ S ₂ O ₃ ¹¹	8 hours unpreserved 28 days preserved ¹¹
Organic Pesticides ¹³	GC and HPLC	EPA (600- Series) ¹³	14	14	14

** Source: 17-160.700, F.A.C.

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- 1. SM XXX procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 16th Edition, 1985.
- 2. P plastic, G Glass
- 3. When specified, sample preservation should be performed immediately upon sample collection.
- 4. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
- 5. The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).
- 6. "Determination of Inorganic Disinfection By-Products by Ion Chromatography, Method 300.0" by John D. Pfaff and Carol a. Brockoff, U.S. EPA, Cincinnati, Ohio 45268 (copy available from the DER QA Section).
- 7. Collect sample in opaque bottles and process under reduced light. Samples on filter taken from water having pH 7 or higher may be placed in airtight plastic bags and stored frozen for up to three weeks. Samples from acidic water must be processed promptly to prevent chlorophyll degradation.
- 8. Temperature and pH must be measured on site at the time of sample collection. Seven days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.
- 9. The eletrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

- 10. Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DER rule 17-3 FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.
- 11. The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with $\rm H_2SO_4$ to pH between 1.5 and 2. Acid-preserved samples, stored at 4° C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.
- 12. DER Central Analytical Laboratory, Tallahassee, FL, Revision No. 1, October 3, 1983. The 1983 draft is available from the DER QA Section.
- 13. Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 643, 644 and 645) which are not included in Table 10 of 40 CFR Part 136 (July 1989).
- 14. Container, preservation and holding time as specified in each individual method shall be followed.

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FIGURE 6.1

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Client				
Sample ID				
Location				
Analysis				
Preservative				
Date	Ву			
Tallahasse (904) 878-		impa, FL i) 885-7427		

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FIGURE 6.2

MONITORING WELL SAMPLING LOG

CLIENT/FACILITY:	
WELL ID;	
WELL LOCKED: YES NO	BAILER PRESENT: YES NO
* WATER LEVEL: (0.01	ft) WELL DEPTH: (ft)
WATER EVACUATION:	(liters) YIELD: (L/H)
FLOATERS: YES NO	_ (ft) SINKERS:YESNO
pH: (units) CAI	LIBRATED: (Date/Time)
** SC: (umhos/cm) CA	LIBRATED: (Date/Time)
TEMP: (6C) CAI	LIBRATED: (Date/Time)
BOTTLES LABELED: YES	NO
SAMPLING COMPLETED:	
BAILER RETURNED & WELL LOCKET	
CUSTODY FORM COMPLETED:	_ YES NO
SAMPLES ICED:YES NO	
COOLERS SEALED: YES	NO SEAL NO:
CARRIER:	DATE/TIME:
COLLECTOR: Signature	DATE/TIME:
NOTES:	
NO.20.	

^{*} Fisher Electronic WL Meter
*** Corning Checkmate 90

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TABLE 6.4

FIELD REAGENT STORAGE AND TRANSPORT

CHEMICAL	METHOD OF STORAGE	METHOD OF TRANSPORT
Nitric Acid	Stored in original container or dedicated repipet dispenser in vented acid storage cabinet; segregated from other acids.	Transferred to dedicated reagent bottle or repipet dispenser; transported in divided box containing only acids (each acid in separate compartment).
Hydrochloric acid	See above	See above
Sulfuric acid	See above	See above
Sodium hydroxide	Dry flake or pellet form stored in original container in reagent cabinet. solutions stored in separate cabinet.	Dry forms transported in original or dedicated transfer container. Solutions transferred to dedicated plastic container and transported segregated from acids.
Zinc acetate solution	Stored in dedicated repipet dispenser in reagent storage cabinet.	Transported in compartmentalized box in capped repipet dispenser.
EDTA Solution	Stored in dedicated repipet dispenser in reagent storage cabinet.	Transported in compartmentalized box in capped repipet dispenser.
Isopropanol	Stored in original container in vented solvent storage cabinet in volatile analysis/custody area.	Transported in bottle jacket in original container.
pH and conductivity standards	Stored in reagent storage cabinet in air conditioned laboratory.	Transported in dedicated plastic containers.

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6.10 Field Waste Disposal

Field-generated wastes will be handled according to the DER SOP General Sampling Protocols, Section VI.B. Wastes transported back to the laboratory for disposal will be handled in accordance with section 8.4 of this document.

6.11 Analyte-Free Water

Analyte-free water used in cleaning and field QC samples is defined as water from any source which exhibits no interferences or analytes of interest above the applicable reporting limits.

Analyte-free water may be obtained from the following sources, but is not limited to these sources.

Laboratory deionized: most inorganics

Laboratory deionized with Milli-Q-type polishing: all analytes

Laboratory deionized with 0.2 micron polishing filler: microbiology

Private well water: any analysis for which acceptability is demonstrated

Purchased deionized: any analysis for which acceptability is demonstrated

Purchased organic-free: VOCs, extractable organics, and any analysis for which acceptability is demonstrated

Analyte-free water will be used as the final rinse in field or lab cleaning procedures, and for trip blanks, field blanks, equipment blanks, and laboratory blanks.

Documentation of analyte-free water sources is maintained via results of trip blanks, equipment blanks, laboratory blanks, control blanks, and container blanks.

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FLORIDA DEPARTMENT OF ENVIRONMENTAL REGULATION Quality Assurance Section

STANDARD OPERATING PROCEDURES TO BE USED AND INCORPORATED BY REFERENCE IN THE COMPREHENSIVE QA PLAN

Name of Organization:
Savannah Laboratories & Environmental Services, Inc.
Address: 5102 LaRoche Avenue Savannah, GA 31404
Comprehensive QA Plan Number: 890142G
Check the specific protocols that your organization will be using while collecting environmental samples. Note: check only documents and protocols as listed in the "DER Quality Assurance Interim Standard Operating Procedures" dated October 29, 1991 for which your organization has current equipment capabilities.
SAMPLING PROTOCOLS X General Sampling X Wastewater X Surface Water X Potable Water X Groundwater X Fish Tissue CALIBRATIONS X Soil X Sediment X Sediment X Surface Waste Sludges X Sludges - Solid and Hazardous Waste
X pH X Dissolved Oxygen X Specific Conductance OVAs X Temperature X Residual Chlorine X Automatic WW Samplers DECONTAMINATION AND CLEANING PROTOCOLS
<pre>X</pre>
X Solid samples - 17-160.700, F.A.C., Table 5 QUALITY CONTROL REQUIREMENTS AND PROTOCOLS Minimum Field quality control requirements

QA Targets for Field Protocols

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STATEMENT OF INTENT TO COMPLY WITH THE STANDARD OPERATING PROCEDURES MANUALS

Janette D. Long Savannah Laboratories (name) Quality Assurance Mana (organization), who were sworn pertinent to the protocols that statement and that these docume Comprehensive Quality Assurance state that the organization of has the equipment and capabili and will require that said pro activity. They state that the Quality Assurance Plan attached the Department's review requir They further state that the in made above are true and correc are aware that any misrepresen of approval of the Comprehensi further constitute violations person making a false oath bef	authority, personally appeared (name) Vice-President (title) (organization), and Alan C. Bailey ger (title) Savannah Laboratories and said that they have obtained copies of all documents they have identified on the opposite side of this ents shall be incorporated by reference into the Plan attached hereto or identified herein. They further which they are officials or officers as identified herein ty to perform the protocols specified by these documents tocols shall be followed when performing the specified y understand that final approval of the Comprehensive d hereto or identified herein is contingent upon satisfying ements in all other sections of the Plan. formation, statements, facts and representations given and to the best of their knowledge and balief, and that they tations or falsifications constitute grounds for rejection ve QA Plan attached hereto or identified herein, and of Section 117.03(2), F.S., which provides that "[a]ny ore a notary public shall be guilty of perjury and shall be res, and disabilities that are prescribed by law in case of
perjury under Chapter 873."	
9-10-92	(Jan I O hong
DATE .	(print name Janette D. Long)
	(Title: Vice-President)
	(Organization Savannah Laboratories)
9-10-92	Mc C. Beiley
DATE	(print name Alan C. Bailey .)
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	(Organization Savannah Laboratories
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7.0 SAMPLE CUSTODY

7.1 Sample Custody Objectives

The primary objective of sample custody is to provide accurate, verified, and traceable records of sample possession and handling from sample container shipment through laboratory receipt and sample disposition.

Evidence of documentation of sample collection, shipment, laboratory receipt and custody is accomplished utilizing a chain-of-custody record (Figure 7.1). A sample is considered in custody if it is:

- in actual possession of the sampler or transferee
- in view after being in physical possession of the sampler or transferee
- sealed so that sample integrity will be maintained while in possession of the sampler or transferee
- in a secured area, restricted to authorized personnel.

7.1.1 Custody Record Maintenance

Field and laboratory records are maintained in a secure area. All field and laboratory data are recorded in bound notebooks and entries are made in waterproof ink. Field and laboratory data entry errors are deleted with a one-line strike through the error. The correction is initialed and dated by the sampling or analytical staff member making the change. Field and laboratory information is documented on prepared forms. All forms for recording field and laboratory data include spaces for date and initials which must be completed by the data recorder. Field and laboratory documentation not recorded on prepared forms is also dated and initialed.

7.2 Sample and Legal Custody Procedures

All samples requiring sample or special legal custody procedures are received by the laboratory custodian under a chain-of-custody procedure. Legal custody is a special type of sample custody in which all events associated with a specific sample are documented in writing.

7.3 Laboratory and Field Custody Procedures

The following procedures apply to the custody activity observed by Savannah Laboratories during sample or legal custody procedures.

7.3.1 Selection and Preparation of Sample Containers Supplied to a Client or Sampling Team

Sample containers provided by SL are constructed from EPA designated materials, contain EPA prescribed preservatives and are affixed with an SL identification label (Figure 7.2). In order to monitor container temperature, a 100-mL plastic container labeled "Sample Container

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SAVANNAH LABORATORIES LENVIRONMENTAL SERVICES, INC.

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ANALYSIS REQUEST AND CHAIR OF CUSTODY RECORD

PROJECT HUMANES

P.O. HUNGER

JUAN INSIDS

CLIENT ADONESS.

MECENTO TON CASONATONY BY, (SIGNATURE)

| 3102 Lefleche Areinia, Sarannah, GA 31104 | 3116 Industrial Plaza Liking, Satahasinea, FL 32001 | 141 Southmass 12th Arenas, Desirbot Brach, FL 32422 | 100 Satahashi Dirin, Walde, At 34633 | 8212 Berlando Mand, Suizo 100, Fange, FL 32431

REQUIRED ANALYSES

LABORATURY HELIANKS

Plant (812) 35 1-7456

Flore: [901] 678-3891 Flore: [303] 431-2400 Flore: [703] 648 6433

Phone: [113] 385-7127

TAGE

STANDAND TAT

FIGURE 7.1

ORIGINAL

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I - FEL OUT REOUEST FORM AND RETAIN PLY 3 (FING COPY) FOR YOUR RE

TRICTIONS

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FIGURE 7.2

SL	SAVANNAH LABI	ORATORIES
\$avannah, GA (912) 354-7858	Deerfield Beach, FL (305) 421-7400	MODHY, AL
Client		
Sample ID	<u>-</u> -	
Location		
Analysis		
Preservative		·
Date		
Tatlahas: (504) 87		amoa, FL 3) 885-7427

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Temperature-Lab Use Only" is prefilled with tap water and supplied with each sample shipment to monitor sample temperature upon receipt.

Projects which require sample containers to be screened for contaminating properties prior to shipment and certified "contaminant-free" can be provided upon the client's request and expense. Containers will be provided with a unique batch assignment number to permit traceability. A sample container preparation logbook (Figure 7.3) is maintained by custody personnel in the event this level of service is requested. All standard custody procedures are maintained for precleaned sample containers.

7.3.2 Chain of Custody Documentation, Traceability, and Sample Integrity

Formal chain-of-custody procedures are initiated by a custody dispatch technician who is responsible for organization and relinquishment of sample containers to the client or field personnel.

All field information must be properly recorded on the chain-of-custody form. Proper completion of the form is the responsibility of the field sampling manager and is required prior to relinquishment of the samples. If the site address is different from the client address, the site address is recorded in the "Project Name" space on the chain-of-custody form, or on the right hand side of the form if additional space is required. The sample identities assigned in the field are recorded in the "Sample Identification" column. Common carriers may identify themselves by signing the "Relinquished By" space on the chain-of-custody form.

For samples transported from the field to the laboratory by common carrier, chain of custody is maintained. Completed custody forms must accompany each sealed cooler, and are placed in a plastic bag and taped to the inside lid of the cooler. Coolers are sealed in the field with the SL Custody Seal (Figure 7.4) or custody tape by the field sampling team to ensure that tampering will be immediately evident. A unique identification number is recorded on the seal and accompanying chain-of-custody form with waterproof ink. A copy of each airbill package tracking form associated with a shipment of samples is maintained in the appropriate client files.

The sample receipt custodian is responsible for the inspection of shipping containers upon laboratory receipt for overall integrity and to ensure that the contents have not been altered or tampered with during transit. If tampering is apparent, the sample receipt custodian immediately contacts the assigned project manager. The sample manager is also notified of the incident and is responsible for client notification. A sample custody excursion form (Figure 7.5) is filed by the sample manager, and any corrective action required by the client is documented on the accompanying project chain-of-custody form which is dated and signed by the sample or project manager.

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Container	Container	Container	Batch	Preservativo	Proservative	St. Project	SL Project	QA Of	ficer	Corrective
Туре	Size	Aperture	Humber	Added	Lot Number	Manager	Number :	Accept	Reject	Action Report Number
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CONTAINER PREPARATION LOG

SL

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FIGURE 7.4

SL SAVANNAH LABORATORIES LENVIRONMENTAL SERVICES. INC.	SCHIM	
OFFICIAL SAMPLE SEAL	344 ML 344	

			SL	SAMPLE C	USTODY 1	EXCURSIO	N		
L Project/SDG #									
Sample Description	Date Sampled	Date Received	Arrival Tomp.	Inappropriate Container	Container Broakago	Container Luakago	Container Label Discrepancy	Conments	Initial
	<u> </u>					,			
	<u></u>			CLIENT N	OTIFICA	rion			
SL Contact	Hotificat	ion Date	Via	Mone/Fax	(lient Contac	t.	Resolution	

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If shipping containers arrive intact, they are immediately opened by the sample receipt custodian in the receiving area, and the chain-of-custody form and temperature container removed for inspection. Container temperature upon receipt is documented in a bound sample registry (Figure 7.6), or if requested by the client, documented on the chain-of-custody form.

7.3.3 Field Custody

When sample collection is performed by SL, Savannah Laboratories' field sampling manager is responsible for ensuring that chain-of-custody procedures for all sampling events are properly documented. The custody forms and login procedures follow the protocol outlined in Section 7.3.

Prior to field sampling, it is preferable to place waterproof sample labels on each sample container and complete each sample label with as much information as possible in waterproof ink. Field sampling technicians are responsible for ensuring that labels are completely filled out upon sampling. Each sample is identified in the field by a unique alphanumeric designation on the label.

All information included on each container label must be included on all field-generated records including: permanent field notebook, individual well log, groundwater elevation form, and chain-of-custody form. This field documentation demonstrates traceability of the containers and samples and links all ancillary records to specific sampling events.

Each sample is packed to ensure against leakage or breakage and to maintain individual sample integrity. All glass containers are secured individually with bubble wrap. Each set of sample containers with the same sample identity is placed together in plastic bags and sealed. When more than one set of sample containers (different sample identities) are placed in the sample cooler, each set must be sealed in a separate plastic bag. All VOA sample vials are wrapped twice in bubble wrap and each set is sealed in a separate plastic bag. Sufficient ice is placed in sealed plastic bags to maintain the sample at 4° C until sample receipt by the laboratory. Additional information regarding sampling can be found in Section 6.0.

Ten percent of samples collected by the SL field sampling team will consist of quality control samples for pH, specific conductivity, temperature, or other client specified parameters per site to satisfy DQO project requirements.

When applicable to the site, the following information is documented by the field technicians in the bound field notebook. This field documentation is reviewed, approved and initialed by the field sampling manager prior to client submission.

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SL	SA	MF	PLE	R	EGISTRY				AQI	JEC	US							NC	ON/	ıQl	JEC	ous							
SL LOG NO.	CUSTODIAN INITIALS .	TIME RECEIVED	DATE RECEIVED	DELIVERED BY	CLIENT ID	מוצפולק היאי ל	L n'm antber glass w/TFE	SO m. m/m plastic	State with smoor glass	250 mt. m/m plastic	250 mL m/m nalgene	125 mL m'm anther glass w/TFE	130 mL mm plastic	43 mL vist w/TFE		L w/m glass	L w/m passic	Story with place		The Mark States	250 mL w/m plentic	125 mL m/m ember glass w/TFE	100 mt. w/m glass	250 m. m/m glass	ADDITIONAL SAMPLE INFORMATION	CONTAINER TEMP.	NOTIFY CLENT	LOGIN COMPUTER	DATE REPORTED
	-	\vdash				-	_	_	_	_	-		_	_	-	-	-	- -	- -	- -	-		-					-	-
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Site location Date/time of sampling Sample identification (including specific location) Sample sequence number Site conditions Weather conditions Purging equipment used Description of QC samples collected Names of personnel/visitors Sampling equipment used Field analysis data Field decontamination techniques Well casing composition and diameter Drilling/boring method Drilling well type/name Water table and well depth Purge volume calculations Volume of water purged Date/time of purging Analytical data to monitor stabilization of well Use of fuel powered units Plumbing/tap material construction Purging flow rate Purging time Flow rate at sample collection Depth samples taken Beginning/ending time for composite sampling Depth soil samples taken Soil sampling technique used Type/description of drums Phases sampled in drums

More complete information is provided regarding sampling procedures and documentation in Section 6.

7.3.4 Sample Documentation, Identification, and Login

A seven-character project code is assigned by division and sequentially in order of sample receipt, recorded on the chain-of-custody form and each sample container submitted with the project and recorded in the bound Sample Registry. Proper and complete sample documentation must be provided on the chain-of-custody form in order to log samples into the sample registry. The sample registry includes all information necessary to maintain chain of custody including laboratory ID, client (field) ID, and initials of the sample receipt custodian. Ancillary information such as sample collection date and requested analyses is transferred directly from the chain-of-custody form into the LIMS, and appears on the client acknowledgement for each project.

Once the chain of custody is verified, the project identified by this unique number is logged into the computerized LIMS (Figure 5.1) to disburse the desired work order request to the laboratory. The sample receipt custodian checks each sample against the chain-of-custody form for

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discrepancies between information on the sample label and information provided on the chain-of-custody form. The sample receipt custodian also inspects all samples for leakage or obvious seal tampering (if provided). All samples are unpacked in a well-ventilated sample receipt area. Personal respirators are provided to each sample receipt staff member for use with any hazardous samples. Samples received in plastic containers which appear to be accumulating or evolving gas are treated cautiously because they may contain toxic fumes or be of an explosive nature.

A space labeled "custody intact" provided on the chain-of-custody form is used to describe the sample condition upon receipt. A "Y" indicates no custody problem was identified and a "N" indicates samples or container integrity was compromised and client notification and corrective action is required.

Discrepancies noted from the custody staff are transmitted to the project and sample manager and are resolved with the client prior to laboratory work assignment. The project manager or the sample manager attempt to resolve custody discrepancies expeditiously to avoid holding time compromises. After a decision concerning a sample has been made, the project manager or sample manager makes an initialed note on the original custody form which states person notified, time, date, and resolution, if applicable.

7.3.5 Sample Preservation

After addition of the project sequential identification number, the samples are dispersed to the appropriate laboratory section sample storage areas. Color-code dots and unique sample bottle types correspond to specific analysis and are stored at designated sample storage areas throughout the laboratory sections. Bound sample storage temperature logs are maintained for all sample storage refrigerators to assure proper temperature maintenance throughout the analytical process.

The color code scheme for the various preservatives used in SL's sample containers is in the Sample Container Request Form which is submitted by a client requesting sample containers. This two-sided form is shown in Figures 7.7 and 7.8.

All sample containers used by the SL field sampling team contain premeasured portions of preservatives. Additional preservatives are obtained prior to each sampling event from parent stocks maintained by the shipping department. Documentation is kept for all additional preservatives used in the field. The effectiveness of pH adjustment by addition of acid or base to the samples is checked after sampling by pouring a small amount of the preserved samples into a small specimen cup and testing with narrow range pH paper. Because of the risk of compromising sample integrity, VOA samples cannot be checked in the field.

FIGURE 7.7

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SAMPLE CONTAINER REQUEST FORM

The number, color-code preservative and container description for the analyses as requested are listed below. A summary of sampling instructions for general analysis categories is referenced on the reverse side.

			.QU	EOL	JS						NOI	VAC	UE	οU:	\$			
																		COLOR PRESERVATIVE CODE
L n/m plastic	L n/m glass w/TFE	500 mL n/m glass	250 ml, m/m plastic	250 mL m/m nalgene	125 mL m/m amber glass w/TFF	100 ml. m/m plastic	40 ml. vial w/TFE			l. w/m glass	t, w/m plastic	500 mt, w/m glass	500 rnL w/m plastic	250 ml. m/m plastic	125 mL in/in amber glass w/TFE	100 ml w/m class		Lab Pk Prep. by: Lab Pk checked by: Quantity of Lab Pks. Shipped: SL Project Mgr.: Sample Coordinator: Comments: Temperature Container
																		NO. OF CONTAINERS SHIPPPED
	-													<u> </u>				NO. OF CONTAINERS/SAMPLE
																		NO. OF TRIP BLANKS
																<u> </u>	1	NO. OF FIELD BLANKS
															<u> </u>	_		NO. OF EQUIPMENT BLANKS
																		GENERAL PARAMETERS
Pho	one N	No: _ Ship	nen	t: _						_								of Shipment:
																		E KEY
RE	D	(A	ľ	nad	e, F	LUS	H IV	ME	DIA	TEL	Υw	ith '	wat	er.				CACID. Avoid skin and eye contact. If contact is
GR	EEN	(G		CAU MM							LFÜ	RIC	AC	ID.	Avc	id	ski	and eye contact. If contact is made, FLUSH
BU	UE	(8)	CAU If co	MO ntac	NI ct is	STF	ON de, F	G C	AUS SH	TIC IMM	I C	ON	TAI	NS will	\$0 1 W	DIL ate	M HYDROXIDE. Avoid skin and eye contact.
TA YE	ITTO/	ie io T W W)))	No p No p Con Con	ores tain tain	erva erva s Zid s Sc NI	ative ative nc A odiu CO	add add ceta m Th	led. led. ate. nios INS	Avo ulfa HY	oid : te. DR(skin Ster	and rilize	d ey	e co	ont:	act	If contact is made, FLUSH IMMEDIATELY with water

DO NOT inhale vapors that may be caused from a chemical reaction between the preservative and sample. Collect sample in a well-ventilated area or use appropriate breathing apparatus. NEVER RINSE sample containers. If skin contact with preservatives occurs, always wash hands IMMEDIATELY.

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GENERAL SAMPLING INSTRUCTIONS

DO NOT PRE-RINSE CONTAINERS. These containers have been specially prepared for specific analyses (See Preservative Color Code Key). Fill container to within 1° of capacity unless otherwise indicated, cap tightly, label and ice. Some requests require multiple containers to perform all analyses. (See Sample Request Form on reverse side.)

LITER PLASTIC

Purple n/m:

Physical Properties. Miscellaneous General (BOD)

Blue n/m:

Cvanide

Red n/m:

Radiological (Rad 226, Rad 228, alpha and beta)

Purple w/m:

Metals and Miscellaneous Inorganics, General, Physical Properties

(Nonaqueous)

LITER GLASS

Purple n/m:

Extractable Organics (BNAs. Pesticides/PCBs, MBAS, Herbicides)

Orange n/m Green n/m:

Dioxin/Dibenzofurans Total Recoverable Phenolics

Purple w/m:

All Organies (excluding Volatiles), Inorganies, Physical Properties, General

(Nonaqueous)

500 PLASTIC

Purple m/m:

Physical Properties, Miscellaneous General

Red m/m:

Metals with Mercury Purple w/m:

(Nonaqueous)

Inorganics. Physical Properties

500 ML GLASS W/TFE

Lt. Blue w/m:

Petroleum Hydrocarbons

Green w/m:

Oil and Grease

Green n/m (amber):

TOX. Fill to capacity.

Purple w/m:

All Organics (excluding Volatiles), Inorganics, Physical Properties, General

(Nonaqueous)

250 ML PLASTIC

Purple nym:

Physical Properties, Inorganics (nutrients), Hexavatent Chromium

Red m/m: Green m/m:

Metals without Mercury Nitrogen series. Phosphorus

Tan ni/m:

Sulfide

250 ML NALGENE

Yellow m/m:

Bacteriological (Coliform, Standard Plate Count) Sterile container - do not touch cap or container

interior. Remove faucet strainer and flush line prior to sample collection.

125 ML AMBER GLASS W/TFE

Green m/m:

TOC. Fill to capacity.

Purple m/m:

Volatiles. Fill to capacity - no headspace.

(Nonaqueous)

100 ML PLASTIC Purple m/m:

Physical Properties, Inorganics (single parameter)

Green m/m:

Nutrients. COD (single parameter)

100 ML GLASS

Purple w/m:

Organics, Inorganics, Physical Properties, General (single parameter)

(nonaqueous):

40 ML GLASS VIAL W/TFE

Lt. Blue n/m:

Volatiles (Aromatics and/or Halogenated constituents). Fill vials until slightly overflowing with

minimum aeration. Place septa W/TFE liner facing sample and seal with NO headspace.

Purple n/m: Yellow n/m: EDB. Volatile Halocarbons. Fill as referenced above. Tribalomethanes (THM). Fill as referenced above.

Container Closure Key (n/m = narrow mouth, m/m = medium mouth, w/m = widemouth)

CONTAINER SHIPPING INSTRUCTIONS

After sample collection, please check all custody forms and sample containers for discrepancies. Sign the custody form and seal in the enclosed plastic bag. To avoid container leakage during transit, additional plastic bags have been included in the shipment to contain ice for sample preservation. Please place these ice bags between the samples and secure the lab pack for shipment. Return lab packs to Savannah Laboratories & Environmental Services, Inc., 5102 LaRoche Avenue, Savannah, GA 31404. If you have any questions concerning containers shipped or acceptable field substitution, please contact your project manager or sample coordinator for assistance at (912) 354-7858 or FAX (912) 352-0165.

Thank you for your patronage.

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All samples received by Savannah Laboratories are checked for proper pH adjustment by the appropriate preparation or analytical department as soon after receipt as possible. The pH of each sample is checked, documented, and adjusted, if necessary. To avoid compromising sample integrity, volatile samples are checked for proper pH adjustment only at the time of analysis. The pH of volatile samples is not adjusted.

7.3.6 Sample Security, Accessibility, Distribution, and Tracking

Only authorized personnel are permitted within the laboratory areas where sample access is possible. Sample storage areas are designed to segregate volatile and nonvolatile samples. Standards and extracts are also departmentally controlled and stored in segregated facilities.

The set of analyses required for a group of samples is project-dependent. After sample login and verification, samples are relinquished from the receiving area to the appropriate sample preparation area. Those samples not requiring preparation are relinquished immediately to the sample analysis storage area. Using LIMS-generated sample preparation worksheets for guidance, samples are extracted, digested, or distilled as appropriate. An example sample preparation log (Cyanide Distillation Log) is shown in Figure 7.9. The extracts, digestates, or distillates are then transferred and relinquished to the appropriate analysis section, where analysis is performed. An example analysis log (Cyanide Analysis) is shown in Figure 7.10.

For projects where in-laboratory custody records are required by the client, the SL project manager should inform the custodian and sample manager to coordinate custody activities prior to sample receipt. For those samples, department-specific in-laboratory sample tracking forms are executed by department staff. An example of a form of this type (Semivolatile Extract Custody Log) is shown in Figure 7.11. Samples and sample preparations are stored in a secure (locked) sample storage area. When samples or sample preparations are removed from or returned to designated storage areas, the form is signed and dated by the analyst.

Sample holding times are tracked via the LIMS. Sample collection dates are routinely entered into the LIMS with all sample logins. This information allows holding times specific to each departmental analysis to be tracked by department managers, supervisors, chemists, and analysts through the use of daily status sheets, reference sheets, and preparation worksheets. Date analyzed is recorded via instrument outputs or analysis forms when applicable as an integral part of the raw data. Upon the analysis of each parameter, the date of analysis is entered into the LIMS and can be compared to the date sampled to validate that holding times have not been compromised.

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FIGURE 7.9

	CYANID	E DISTILLATION LOG	
Spike Date Check	Level Stock CN Prepped Standard	Date Analyst Batch # Final Volume	
#	SL Log #	Sample Description	Wet Weight
	Blank		
	ERA		
	· · · · · · · · · · · · · · · · · · ·		
			
			
			-
		· · · · · · · · · · · · · · · · · · ·	
	·		
			-
			
-			

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FIGURE 7.10

		CYAN	IDE ANA		oG			
	STANDA	RD CURVE		Date				
	mg/L	Å		Analyst			·	
	0.50			Batch #				
	0.30 0.10			ł		efficient		
	0.070 0.040			QC Check	Tr	ue Value		
	0.010							
*	SL Log #	Sample Description	Sample Dilution	Volume or Wet Weight	Sample Å	Result mg/L	Dry Weight	Result mg/kg
-								
<u> </u>								
⊩			<u> </u>	<u> </u>				
	!					<u> </u>	<u> </u>	
-								
-								
<u> </u>								

FIGURE 7.11

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			,		 	 	 ,	,		 	 	 ·····
	Date Disposed	:										
	Instrument .	ì										
	Received	Date										
N LOG	Rec	Int										
CUSTOE	thed from ction bent	Date										
CTRACT	Relinquished from Extraction Department	Int										
SEMIVOLATILE EXTRACT CUSTODY LOG	Date Complete (Final	Proj Clik)										
SEMIVOI	Analysis				٠							
SI	Box ID							_				
	Total # of Extracts											
	# 20											

"-EXTCUS.LOG:09.10.92.0

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7.3.7 Sample Disposition

After analysis completion, custody of unused sample portions, extracts, or digests is relinquished to the central secured storage area. Unless a client requests the project manager to save unused samples, digests, or extracts, disposal from the central storage occurs as soon as holding times have expired or three weeks after results submission.

Requests for extended sample, digest or extract storage must be provided by the client to the SL project manager in writing (or contract form) prior to sample receipt and extended storage will usually result in additional fees to be negotiated by the SL project manager prior to sample receipt. SL is not responsible for evaporation or other deterioration of samples, extracts, or digests during extended storage periods.

Prior to report submission, the project manager reviews all analytical results, and if the results reveal that a sample is hazardous per 40 CFR Part 261 characteristics or contains 50 ppm or greater PCBs; or if client-supplied information (chain-of-custody forms, etc.) states that the sample is hazardous; or if client's instructions or a contract requires all samples be treated as hazardous waste, the project manager will arrange for samples to be returned to the client or disposed of per client's instructions at the client's expense. Tracking and disposal of hazardous samples is accomplished and documented via the LIMS system.

7.3.8 Interdivisional Custody

The laboratory director at each location monitors the sample load and turnaround time through LIMS-generated reports. If it appears that analysis demand will exceed capacity, or if instrument failure occurs, samples may be transferred (provided client contracts or arrangements, project QA plans or certification limitations do not prohibit sample transfer) to another SL division to ensure that holding times and turnaround commitments are met.

If samples are transferred to another division laboratory, full custody is maintained. Special determination codes specific to each laboratory location are entered into the LIMS to enable the project manager and laboratory director to track sample progress and maintain chain of custody. Copies of the original chain-of-custody form (executed for interdivisional sample submittal), computerized LIMS work order acknowledgements, and extract or digest preparation logs pertinent to the project order accompany the samples or preparations. This material includes dates of sample preparation and requested analyses. Upon sample receipt at the other division laboratory, standard custody procedures are followed.

7.4 Electronic Data Records

By careful assignment of user passwords and file access/lock codes, Savannah Laboratories maintains a high level of data security for the LIMS. Thus, only authorized SL personnel can access client files to view

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data. In addition, data entry and editing is restricted to highly trained data management personnel.

If requested, data can be electronically transferred to the client via modem. To insure data integrity, the specific client's data are first downloaded to an off-line PC and then electronically transferred to the client. Access to the PC via modem is controlled by assignment of a confidential password to the client.

Signed hard copies of reports and not electronic or diskette deliverables are the official report and are always submitted to clients who request electronic data transfer, which allows verification of downloaded information. SL is not responsible for electronic transfer or diskette errors and maintains that it is the client's responsibility to check all electronic or diskette data against the hard copy. A download information file is maintained by the LIMS manager.

Internal documentation is maintained by the LIMS manager for all LIMS programs. This documentation includes descriptions of any program additions, deletions, or modifications, the date of revision, and the initials of the responsible programmer. To verify proper program functioning of the hardware and software, a simulation account is maintained. When hardware/software modifications are made, this account uses actual data to model an account in order to verify the modifications are functioning as anticipated. Antivirus software serves the LIMS as a protective measure.

At present, laboratory instrumentation is not interfaced directly to the LIMS and thus, no instrument-LIMS data transfer step requires verification. All instrument data is verified by chemists or analysts as described in Section 12.5.2.

Entry of data into the LIMS from chemists' worksheets is performed three times weekly by data entry technicians. Immediately following data entry, approval sheets are printed with the entered data as it appears in the LIMS. Assistant project managers compare all data on the approval sheets, versus the chemists' worksheets for data transcription errors.

7.5 Verification of Hard Copy Records

Forms that are routinely printed for verification and signatures include data worksheets, data approval forms, and final reports. Hard copies of final reports, field data, chain of custody forms, and any ancillary documentation pertinent to the project will be stored in a secured storage area and placed in files alphabetically by client and chronologically within each client folder.

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8.0 ANALYTICAL PROCEDURES

The ultimate responsibility for analytical method selection lies with the client or regulatory agencies. Whenever possible, laboratory and field analysis of all samples are conducted by EPA-approved methodology. When EPA approved methods do not exist or project protocols require alternative methods, these methods must be approved by the client and the appropriate regulatory agency.

Tables 5.1 and 5.2 list Savannah Laboratories' routine laboratory parameters with their respective method numbers. Table 5.3 lists field parameters with their respective method numbers.

A detailed SOP has been prepared for each routine analytical method. Copies of SOPs are kept at the respective analytical benches, or by each department/section supervisors, and the QA manager or laboratory director.

In cases where GC, LC, or GC/MS methods are used to determine compounds not included in the actual method list, these unlisted parameters are flagged in the tables with a triple asterisk (***) and method validation data are included in Appendix A.

For those cases where no specific soil or sediment method exists, water methods are adapted. These adaptations are described in Section 8.2, and validation data are presented in Appendix A. Unless indicated in the appropriate SOP, all parameters listed in Tables 5.1 through 5.3 are analyzed by the methods referenced, without modifications. Interpretation of ambiguous method requirements is accomplished by consulting with regulatory agencies and EPA laboratory/QA personnel.

8.1 Glassware Cleaning Procedures

Laboratory glassware washing procedures are adapted from SW-846, 40 CFR Part 136, Standard Methods, and EPA 600/4-79-019, and are as follows:

Extractable Organics

Prerinse each item with the solvent to be used in it. As soon as possible after use, rinse with lab-grade acetone. Wash with hot water and a nonphosphate detergent such as Alconox, scrubbing thoroughly with a brush. Rinse thoroughly with tap water at least three times. Rinse inside surface with Nochromix solution, catching rinsate for re-use. Rinse again with tap water, followed by pesticide-grade acetone. Rinsing with hexane is avoided to minimize the possibility of contamination of glassware used for total petroleum hydrocarbon determination. Air dry when possible, and do not bake Class A volumetric glassware. Store glassware inverted or cap openings with foil to exclude dust and other contaminants. Because of possible damage, caps, septa, and plastic items are not rinsed with Nochromix.

Volatile Organics

Wash with tap water and Alconox or Liquinox, then rinse thoroughly with organic free water. Oven dry at 110°- 120°C for at least two hours. Do

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not bake Class A volumetric glassware. Glassware is usually stored in the oven until use. Caps and septa are washed in the same manner, but caps are not oven-dried. Highly contaminated glassware is allowed to soak in Nochromix solution overnight, then washed as above.

General Chemistry, Microbiology, Nutrients, Demands

Wash with hot tap water and Liquinox, rinse thoroughly with tap and deionized water, and air dry. Store glassware inverted or cap openings with foil. Autoclave bacteriological laboratory glassware and collection bottles as described in analytical procedures. COD digestion tubes and caps are cleaned with brushing and tap water (no soap) and rinsed thoroughly with deionized water. Tubes for TKN and total phosphorus sample digestions are washed with hot water and Liquinox, and rinsed with tap water, Nochromix, and deionized water.

Metals/Radionuclides

Wash glass, plastic, and Teflon items in hot tap water and Alconox. Rinse with tap water, 1:1 nitric acid, tap water, and deionized water. Teflon beakers used for sample digestion are further decontaminated by adding 20 mL nitric acid and 12 mL hydrochloric acid, covering with a watch glass, and digesting on a hot plate for two hours. Following this treatment, they are rinsed with 10% nitric acid and deionized water and allowed to air dry.

8.2 Soil Sample Preparation Notes

In the absence of an approved soil method, water methods are adapted for soil matrices. The following soil preparation procedures are applied to parameters in Table 5.2.

1. Fluoride (extractable): Method 340.2

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle, the supernatant decanted, and the extract is analyzed as a liquid sample.

2. Alpha and Beta Radioactivity: Method 9310/900

Soil is ground to a fine powder with mortar and pestle, and 50 to 100 mg soil is weighed onto a tared planchet. Sample is distributed evenly over planchet surface, fixed with clear acrylic solution, dried, and counted.

Chloride (extractable): Method 9251/407A

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant is decanted. The extract is analyzed as a liquid sample.

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Sulfate (extractable): Method 9036/9038/375.3

Approximately 5 g of sample is weighed out exactly and placed in a 100-mL screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the extract is filtered using a syringe filter with a 0.20-um pore size filter and analyzed as a liquid sample.

5. Orthophosphate (extractable): Method 365.1

Approximately 5 g of sample is weighed out exactly and placed in a screw-cap plastic bottle. One hundred mL of DI water is added to the sample, the bottle is capped and placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant is decanted. The extract is analyzed as a liquid sample.

6. Surfactants: Method 425.1

Sample (10-20 g) is weighed out exactly into a 500-mL screw-cap bottle. A volume of water equivalent to 20 times the sample weight is added to the sample, the bottle is capped, placed in a rotating extractor, and rotated for 2 hours. Upon removal, the sample is allowed to settle and the supernatant is decanted. The extract is analyzed as a liquid sample.

8.3 Deviations from Referenced Analytical Methods

In the determination of sulfide in liquid samples containing turbidity or color and in all soil or sediment samples, samples are distilled as per SW-846 method 9030. Upon distillation of the sample, the trapping solution is analyzed colorimetrically as a clear liquid sample as per EPA method 376.2.

8.4 Reagent Storage and Documentation

Reagents are stored with consideration for safety and maximum shelf life. Storage conditions for various classes of reagents are given in Table 8.1, as well as discussed below. Documentation maintenance status for the reagent classes is also given in Table 8.1.

All acids, except those poured up in small marked containers which are for immediate use, are stored in the original containers in acid storage cabinets.

All bases, except those poured up in small containers for immediate use and those that are standardized for specific purposes, are stored in the original containers in designated areas or storage cabinets.

All flammable solvents, except those poured up for immediate use are stored in original containers in approved vented flammable storage cabinets which are located in air conditioned areas.

Dry reagents are stored in designated cabinets in cool, dry areas. Reactive chemicals, cyanides and sulfides are labeled and isolated from other chemicals.

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All acids used for metal sample digestions and all solvents used for semivolatile sample extraction are tested prior to initial use. Specific acceptable chemical lots are reserved and stored by the vendor(s) and are requisitioned and received as needed by the laboratory. Lot numbers used for digestions or extractions are recorded in bound notebooks in the appropriate departments.

Reagent blanks are analyzed with each sample batch for all methods, validating the purity of all reagents. All reagent containers are dated when received, and dated and initialed when opened (except high use items consumed in less than one week). Documentation is maintained to provide traceability of the reagents used with the analysis of any batch to specific reagent lot numbers.

8.5 Waste Disposal

All waste disposal is carried out in accordance to Savannah Laboratories' Waste Disposal SOP. This document includes procedures for identification, storage, personnel training, tracking forms, report forms, safety, as well as details of the disposal. Hazardous waste disposal procedures are given in Table 8.2 and discussed below.

Hazardous wastes must:

- -- be disposed of prior to accumulation of 1,000 kg (approximately 5 drums) of hazardous waste or 100 kg (0.5 drums) of acutely hazardous waste (261.33 (a) (e) P list).
- -- Be generated at a rate of less than 100 kg of total hazardous waste per facility per month (or 1 kg of acutely hazardous waste).
- -- be stored in non-leaking containers in good condition with closefitting lids and kept closed when wastes are not being added or removed.
- -- be accurately labeled with waterproof labels. Labels must specify the words "Hazardous Waste", the composition and physical state of the waste, the hazardous properties of the waste (e.g., flammable, reactive, etc.), and the name and address of the generator.
- -- be clearly labeled with the date that the period of accumulation began on each container and the Hazardous Waste Tracking Log Form.
- -- be handled in containers and in a way that minimizes the possibility of spills and escape of wastes into the environment.
- be stored in an area which is regularly inspected for deteriorating or leaking containers.

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All waste must be segregated for temporary accumulation and storage as well as for disposal. Care must be taken to combine waste materials into categories or waste streams based upon their compatibility.

The following four types of waste are stored in 55-gallon drums.

- Halogenated solvents (methylene chloride and others) -- Store in closed cap metal drum)
- 2. Freon -- Store in closed cap metal drum
- 3. Nonhalogenated flammable solvents -- Store in closed cap metal drum
- Heavy metals or other aqueous wastes (except cyanide) -- Store in poly drum

All other wastes should be stored in the original container or 4-liter glass bottles and disposed of via lab pak. (Packed by disposal company in 55-gallon open top drums.)

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9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Laboratory Equipment

Savannah Laboratories is equipped with state-of-the-art instrumentation to provide quality analytical data to clients. A list of the instrumentation maintained by Savannah Laboratories for the determination of the parameters contained in Tables 5.1 and 5.2 is found in Table 9.1. A list of all field instrumentation maintained by the laboratory is contained in Table 9.2.

9.2 Standard Receipt and Traceability

Standards are purchased from commercial sources in mixes designed for the specific methods or as neat compounds. Certificates of analysis are shipped with each ampule by the vendor. The standards are certified to meet or exceed the criteria established by the U.S. EPA.

Upon receipt, dates are placed on all standard materials. Standard logbooks are maintained by all sections of the laboratory to document the traceability of working standards back to neat materials or prepared stock mixes. All standards are assigned a lot number that provides a unique identification as well as identifying the type of standard (i.e., working). This unique lot number is documented in a laboratory notebook along with date of preparation, initials of preparer, concentration, expiration date (if applicable), and solvent (if applicable). A standard preparation narrative is also provided in this notebook to detailing the preparation steps for each standard.

9.3 Standard Sources and Preparation

Savannah Laboratories maintains an inventory of materials to produce stock standards or purchases stock standards from commercial vendors. Laboratory preparation of all lab-prepared stock, intermediate, and working standards is documented by the responsible analysts. Table 9.3 presents standard sources and preparation protocols for various sections of the laboratory. Field instruments requiring calibration standards (conductivity meters and pH meters) use the same sources as laboratory instrumentation.

Table 9.4 lists titrants used by the laboratory and information regarding their standardization.

9.4 Laboratory Instrument Calibration

The calibration procedures given below meet or exceed EPA method requirements.

Any method calibration requirements which are more stringent than these procedures will be used.

TABLE 9.1

•	Instrument	Deerfield Beach	Tallahassee	Savannah	Hobile .	Tempa Bey
6	ICP Units	1-Jerrell Ash 61	1-Jarrell Ash 61	1-Jarrell Ash 61 1-Jarrell Ash Enviro 36	1-Jarrell Ash 61E	1-Jarrell Ash 61E
5	Mercury Cold Vapor Units	1-Varian VGA/AA20	1-Varian VGA-76/AA20	1-Verian VGA-76/AA20	1-Perkin Elmer 5000	1-Coleman 50B
15	Atomic Absorption Furnace/Flame	1-Varian 400Z 1-Perkin Elmer 2380	2-Varian 400Z 1-Verian AA 20	2-Varian 400Z 2-Jarrell Ash 22/4000 1-Perkin Elmer 2380	1-Verien 400Z 1-Perkin Elmer 5000	1-Varien 400Z 1-Varien AA 20
10	GC/MS Semivolatiles	1-HP 5970	2-HP 5970	5-HP 5970	1-HP 5970	1-HP 5971A
10	GC/MS Volatiles	1-HP 5970	2-HP 5970	3-HP 5970 2-HP 5971	1-HP 5970	1-HP 5971A
32	Gas Chromatography Samivolatiles	3-Varian 3400 with dual ECD 3-Varian 3400 with dual FID	1-Varian 3400 with NPD/ECD 1-Varian 3400 with dual NPD 2-Varian 3400 with dual FID 3-Varian 3400 with dual ECD 1-Shimadzu 9AM with dual ECD 1-HP 5880 with FID	1-Varian 3400 with dual FID 1-Varian 3400 with quad FID 1-Varian 3400 with dual NPD 2-Varian 3400 with dual ECD 2-Varian 3700 with dual ECD 1-Varian 3700 with ECD	2-Varian 3400 with dual ECD 1-Varian 3300 with dual FID 1-Varian 3300 with dual NPD	2-Varian 3400 with dual ECD 2-Varian 3300 with dual FID
24	Gas Chromatography Volatiles/P&T	1-Varian 3600 with PID/Hall 3-Varian 3300 with PID/Hall 1-Varian 3300 with PID/FID 1-Varian 3300 with FID/Hall	1-Varian 3700 with Hall/FID 1-Varian 3300 with Hall/FID 1-Varian 3300 with PID/Hall 1-Varian 3400 with PID/Hall 1-Varian 3400 with PID/Hall 1-Varian 3600 with PID/Hall 1-Varian 3600 with PID/Hall	1-Varian 3700 with Hall/PID 1-Varian 3700 with Hall/FID 1-Varian 3600 with Hall/PID 1-Varian 3400 with PID/FID 2-Varian 3300 with Hall/FID	1-Varian 3300 with Hall/FID 1-Varian 3300 with Hall/PID	1-Verien 3300 with FID/Hell 1-Tracor 540 with PID/Hell 1-Verien 3600 with PID/Hell 1-Verien 3400 with FID/PID
3	TOC Analyzers	1-OI 524		1-Dohrmann DC80	1-01 524	

TABLE 9.1

•	Instrument	Deerfield Beach	Tallahazsee	Savannah	Mobile	Ташра Вау
6	IR Spectrophotometers	1-Perkin Elmer 710	1-Perkin Elmer 727	1-Perkin Elmer 710 1-Buck Scientific HC-404	1-Buck Scientific HC-404	1-Buck Scientific HC-404
7	UV-VIS Spectrophotometers	1-B&L 21	1-Milton Roy 301 1-B&L 21	1-Milton Roy 301 1-Perkin Elmer 35 1-8&L 88	1-Sequoia Turner 340	1-Milton Roy 301
3	Nutrient Autoenalyzers			3-Technicon Traacs 800		
4	HPLC Units	1-Waters 501/481	1-Waters 484 1-Kratos 980 1-Waters 490E			
1	Alpha/Beta					1-Tennelec 5100
1	Scaler					1-Ludlam Measurements 2000
1	Radon Flask Counter					1-Ludlam Measurements 182
1	Ion Chromatograph			1-Dionex 2010		-
4	DO Meters	1-YSI 50B	1-YSI 50B	1-YSI 50B	1-YSI 58	1-YSI 50B
5	Turbidimeters	1-Orbeen-Hallige	1-Hech 1680D	1-Hach 2100A	1-Hach 2100A	1-Hach 1680D
2	TOX Analyzers			2-Dohrmann MC-3		
2	Conductivity Meters	1-YSI 35	1-YSI 33	1-YSI 35	1-YSI 32	
1	Bomb Calorimeter			1-Parr 1341		
15	pH/ISE Maters	Various Orion and Fisher Meters	Various Orion and Fisher Meters	Various Orion and Fisher Meters	Various Orion and Fisher Maters	Various Orion and Fisher Meters
7	Analytical Balance	1-Mettler AE163	1-Mettler AE160	1-Mettler ME160 1-Mettler AE200	1-Mettler AE160 1-Sartorius 1602	1-Mettler AE100

TABLE 9.1

MAJOR LABORATORY INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION							
+	Instrument	Deerfield Beach	Tellahassee	Savannah	Mobile	Tampa Bay	
18	Top Loading Salance	2-Sartorius	1-Mettler PE1600 1-Sertorius L2200-S 1-Sertorius PT1200	1-Mettler PM 2000 2-Sartorius GMBM 2-Mettler 1600 PE 1-Fisher XL500 1-Sartorius 1202	1-SP DTL4100 1-Sertorius B3100P 1-Sertorius FT600	3-Mettler PM3000	
5	Autoclave	1-Napco 8000-DSE	1-Napco 9000D	1-Napco 9000D	1-Napco 9000D	1-Napco 9000D	
15	Waterbath	2-Fisher Versabeth	2-Fisher 1-Baxter	1-C-M Equatherm 2-Fisher Versabath 1-Fisher 20L	1-Fisher Scientific 1-Baxter Durabeth 1-Blue M Magi-Whirl 1-SPB7001-2	1-Branson 3200 1-Baxter Durabath	
6	Biological Incubator	1-Bexter Tempcon	1-Blue M Stabil- Therm	1-Leb-Liner 3554-17	1-Fisher 630D 1-Precision Coliform	1-Blue H	
6	BOD Incubator	1-Westinghouse 16.8	1-Precision 815	1-Precision Lo-Temp	2-Precision Lo-Temp	1-Fisher 307	
20	Drying Oven	1-Fisher Isatemp 500	1-Fisher Isotemp 500 3-Blue M 2-Fisher Isotemp 655G 1-Tempcon	4-Fisher Isotemp 500 1-Tempcon N8620-1 1-Blue M	3-Blue M 1-Precision Scientific	3-VWR 13054	
8	Block Digestor	1-Hach	1-Thermolyne Dri- Bath	2-Tachnicon BD-40 1-Thermolyne Dri-Bath 1-Lab-Line Multiblank 2093	1-Techni: Dri-Block DB-3H	1-Thermolyne	
5	TCLP (nonvolatile)	SL Custom	SL Custom	SL Custom	SL Custom	SL Custom	
2	TCLP (ZHE)			1-ATCS ZHE	1-ATCS ZHE		

TABLE 9.2

MAJOR FIELD INSTRUMENTS AT EACH SAVANNAH LABORATORIES LOCATION

	Instrument Deerfield Beach		Tallahazzee Savannah		Mobile	Тетра Вау	
4	pH/SC/DO/T* Heters	1-Corning Checkmate 90	1-Corning Checkmate 90	1-Corning Checkmate 90		1-Corning Checkmate	
3	pH/Temp Meters		1-Orion 23A	1-Orion SA-230	1-Orion 23A	1-Orion 23A	
3	Conductivity Meters	1 YSI 33	1-YSI 33	1-YSI 33	1-YSI 33		
2	DO Meters		1-YSI 51B	1-YSI 51B	1-YSI 50B		
2	Turbidimeters		1-Hach 16800	1-DRT 15C			
2	Water Level Meters		1-Slope 51453	1-Fisher			

TABLE 9.3

STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standerd Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
ICP	Baker/Spex	Stock 1,000 or 10,000 ppm solutions	Room temp	Working std prepped directly from stock	Room temp	Quarterly or as needed
AA	Baker/Spex	Stock 1,000 ppm solutions	Room temp	Intermediate stds prepped from stocks. Working stds prepped from intermediates.	Room temp	Biweekly Weekly
Autoanelyzer	Fisher Baker	Neat material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
Ion Chromatograph	Fisher Baker Mellinckrodt	Neat material	Room temp	Stock stds prepped from solids, Intermediate stds from stocks, Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
UV-VIS Spectrophotometer	Fisher Baker EM	Neat Material	Room temp	Stock stds prepped from solids. Intermediate stds from stocks. Working stds from intermediates.	Refrigerator Used immediately Used immediately	Monthly Daily or as needed Daily or as needed
IR Spectrophotometer	Fisher	Neat liquids	Room temp	Stock std prepped from nest liquid, Working stds from stock,	Refrigerator Refrigerator	Monthly Monthly

TABLE 9.3

STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Frequency
Turbidimeter	Hach	Standard 4000 ppm formazin solution	Refrigerator	Working stds prepped from stock.	Used immediately	As needed to check Gelex stds
Conductivity Meter	YSI or Fisher	Stendard solution or neat KC1	Room temp	Used as is or prepare from neat.	Room temperature	As needed
тос	Mallinckrodt	Neet KHP	Room temp	Stock std from solid Working std from stock.	Refrigerator Refrigerator	Monthly As needed
pH Meter	Fisher	Calibration buffer solutions	Room temp	Used as is.		
ISE	Baker	Neat material	Room temp	Stock std from source. Intermediate std from atock. Working std from intermediate.	Refrigerator Refrigerator Used immediately	Monthly Monthly or as needed As needed
TOX	Fisher	Neat material	Room temp	Std from source,	Room temp	Monthly
Bomb Calorimater	Parr	Neat teblets	Room temp	Used as is.		

TABLE 9.3

STANDARD SOURCE AND PREPARATION FOR LABORATORY INSTRUMENTATION

Instrument Group	Standard Source	How Received	Source Storage	Preparation From Source	Lab Stock Storage	Prep Fraquency
Gas Chromatographs and GC/HS (Volatiles)	Supelco, Ultra, Accustandard, ChemService, Bexter, Aldrich	Neat Solutions (50-2000 ppm)	Freezer	Stock stds from neat sources. Intermediate stds from stocks. Working standards from intermediates and/or purchased solutions.	Freezer Freezer Freezer	Annually or as noted by manufacturer expiration date. Semiannually (2 months or sooner for gases, styrene, 2- chloroethylvinyl ether) Weekly
Gas Chromatographs and GC/MS (Semivolatiles)	Restek, ChemService, Crascent Chemical, Aldrich, Ultra	Neat Solutions (50-10000 ppm)	Refrigerator	Stock stds from neat sources. Intermediate stds from stocks. Working standards from intermediates.	Refrigerator or freazer Refrigerator or freezer Refrigerator or freezer	Semi-annually or annually as required Semi-annually or annually as requied Semiannually or as needed
High Performance Liquid Chromatographs	ChemService, Crescent Chemical, Supelco	Neat Solutions > 1000 ppm	Refrigerator	Stock stdm from neet sources. Intermsdiate stds from stocks and/or purchased solutions. Working stenderds from intermediates.	Refrigerator Refrigerator Refrigerator	Semi-annually Monthly Weekly

TABLE 9.4

STANDARDIZATION OF TITRATING SOLUTIONS

Analysis	Solution Requiring Standardization	Standard Identity	Standard Source	Frequency of Standardization
Acidity	Sodium Hydroxide (0.02 N)	KHP	Mallinockrodt	With each batch
Alkelinity	Sulfuric acid	Na,CO,	Mallinckrodt	With each batch
COD	Ferrous ammonium sulfate	K,Cr,O,	Mallinckrodt	With each batch
Chloride	Silver nitrate	NaCl	Baker	With each batch (or purchased certified)
Sulfide	Sulfide working standard	I,/Na,S,O,	VWR/Baker	Weekly
TOC (Soil)	Ferrous sulfate	I,/Na,S,O,	VWR/Baker	With each batch

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9.4.1 Metals

ICP

The inductively coupled plasma atomic emission spectrophotometer is standardized daily with single concentration standard solution containing metals of interest and a blank. After calibration, ICV standards are analyzed and must agree within \pm 10% of true value. A blank is then run and must be below the PQL. A 2-5XIDL solution is then analyzed. This is followed by interference check standards A and AB which must be within \pm 20% of true values. CCV standards are run after every 10 samples and sample data must be bracketed by calibration verification standards that are \pm 10% of true values in order for data to be acceptable. Duplicate lab control standards are digested and analyzed with each batch of sample to determine accuracy and precision, and must be recovered 80-120% for liquid samples and 70-130% for soil samples.

AA

Furnace atomic absorption spectrophotometers are calibrated daily with a minimum of three standards and a blank. An initial calibration verification standard is analyzed immediately upon calibration, and must meet accuracy criteria of 90-110%. The initial calibration blank is analyzed, and must be less than the PQL. Lab control standards (digested standards) are analyzed in duplicate for every batch of 20 samples and must be recovered within 80-120% for liquids and 70-130% for soils for the batch to be acceptable. Calibration verification standards are analyzed after every 10 samples and must be recovered within 80-120% for bracketed data to be acceptable.

9.4.2 General Chemistry

Autoanalyzer

A calibration curve containing a minimum of five points is analyzed at least daily. The correlation coefficient from application of linear regression to these points must be ≥ 0.995 . Independent calibration verification standards and blanks are analyzed immediately following the calibration standards and thereafter, after every 10 samples. The initial calibration verification must be within accuracy control criteria given in Table 5.1 or 5.2 for any data to be acceptable. All data must be bracketed by calibration verification standards that meet all criteria given in Table 5.1 or 5.2 for that data to be acceptable.

Ion Chromatograph

For initial validation of the method and to determine linearity of the calibration curve, three to five standards are analyzed. Either linear regression or quadratic curve fitting is used, depending on analyte. The linear regression correlation coefficient must be > 0.990 for any analyte to be considered as giving a linear response. After initial validation, for linear analytes, the instrument is standardized daily with a single point standard. Calibration verification standards are analyzed immediately upon calibration and thereafter, after every 10 samples. The

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calibration verification standards must be within control criteria given in Tables 5.1 or 5.2 to be acceptable.

UV-VIS Spectrophotometer

The spectrophotometer is calibrated at least daily with a minimum of five standards. Linear regression is used to find the calibration curve. The correlation coefficient must be > 0.995 in order for the curve to be acceptable. Calibration verification standards are analyzed immediately following the calibration standards and after every 10 samples. The calibration verification standards must meet control criteria given in Tables 5.1 or 5.2 in order for bracketed data to be acceptable.

IR Spectrophotometer

The infrared spectrophotometer is calibrated daily with a minimum of five standards. The curve is found by linear regression, and the correlation coefficient must be > 0.995. A calibration verification standard is analyzed immediately upon calibration, and after every 10 samples. Calibration verification standards must meet control criteria given in Tables 5.1 or 5.2 in order for bracketed data to be acceptable.

Turbidimeter

Gelex solid standards are calibrated against formazin standards initially and then quarterly. Then, the instrument is calibrated daily with one Gelex standard for each range of interest. A mid-range calibration verification is analyzed for every 10 samples and must meet control criteria specified in Table 5.1.

Conductivity Meter

The cell constant of each meter is determined at a minimum annually by the analysis of five KCl standards. To verify the cell constant, a verification standard is analyzed at the beginning of each working day, using a KCl standard in the expected range of the samples. For meters not having automatic temperature compensation, all samples are analyzed at 25° C \pm 2° C.

pH Meter

The pH meter is calibrated daily with two standard buffers at pH 7.0 and either 4.0 or 10.0, and checked with a third buffer at 10.0 or 4.0 which must indicate \pm 0.10 pH units of its given value. A calibration verification standard is analyzed immediately upon calibration and after every 10 samples. The calibration verification standard must meet criteria given in Table 5.1 in order for bracketed data to be acceptable. Manual or automatic temperature compensation is performed, depending on the meter.

TOC

A single point standard is used to calibrate the instrument daily. A calibration verification standard is analyzed immediately upon calibration

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and after 10 samples. The calibration verification standards must meet control criteria given in Table 5.1 in order to accept bracketed data.

ISE

Ion selective electrodes are calibrated with a minimum of five standards. Linear regression applied to a plot of the log of the standard concentrations versus potential must result in a correlation coefficient > 0.995. Calibration verification standards are analyzed immediately upon calibration and after every 10 samples, and must meet control criteria given in Tables 5.1 or 5.2 in order for bracketed data to be acceptable.

TOX

Although the TOX instrument provides an "absolute" measurement, and is not subject to calibration, a check standard is analyzed daily immediately after the blank, and must meet control criteria given in Tables 5.1 or 5.2 in order for data to be acceptable.

Bomb Calorimeter

The energy equivalent of the bomb calorimeter is determined quarterly by bombing six standard benzoic acid tablets. A fuel oil standard is analyzed in duplicate for every batch of samples, and must meet control criteria given in Table 5.2 in order for data to be acceptable.

DO Meter

DO meters are calibrated prior to use either by Winkler titration or the air calibration technique, and annually by Winkler titration.

Temperature

All laboratory and field thermometers are calibrated annually by comparison with a NIST-certified thermometer. Field meters with automated temperature compensation are checked before use with a calibrated thermometer.

9.4.3 Gas Chromatographs

Volatiles

Initial calibration is performed upon instrument startup and whenever continuing calibration fails the acceptance criteria. A five-point standard curve is prepared using all target compounds. The low standard concentration is near the PQL, and the high standard defines the usable linear range of the detector. After the five standards are purged and analyzed, a calibration curve is generated using internal standard methodology. If the internal standard exhibits matrix interference in sample, external standard methodology may be used; however, an internal standard is preferred for purge-and-trap methods. Ideally, all volatile compounds should exhibit enough linearity to use a straight line fit forced through the origin. However, some compounds may exhibit true non-linearity but consistent performance using a quadratic fit. A quadratic

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fit curve may be used. The analyst should visually inspect the curves before proceeding with sample analysis.

An alternative to quantitation from a calibration curve is quantitation from an average response factor (RF). This is an acceptable technique for all SW-846 8000-series methods, all 40 CFR 136 600-series methods, and all 500-series drinking water methods. For the 8000-series methods, if the % RSD is < 20%, the average RF may be used. For the 500- and 600-series methods, if the % RSD is < 10%, the average RF may be used. Quantitation from the curve is preferred.

Continuing calibration check (CCC) standards are analyzed at the intervals specified in the methods. The CCC standard concentration is normally the mid-point of the five-point calibration curve, and must be at the level specified in the method "Q-tables" for the 600- and 8000-series methods. The 500- and 600-series methods specify a mid-level CCC at the beginning of each working day. The 8000-series methods specify a mid-level standard at the beginning of each working day and after every ten samples thereafter if needed for further sample analyses. The acceptance criteria for the 600- and 8000-series methods for volatiles are listed in each method's "Q-table." The analyzed value of each standard component must fall within the range of values given in the table. For compounds not present on the Q-table, the analyzed value must fall within 15% of the true value, or the laboratory may generate internal acceptance ranges based on a minimum of thirty data points. The acceptance limits for the 500-series methods are ± 20% of the true value.

If the CCC standard fails acceptance criteria, another CCC standard may be analyzed. If the second standard also fails, the initial calibration must be repeated.

2-Chloroethyl vinyl ether exhibits erratic chromatographic behavior. The Supelco, Inc. Purgeable A Mixture footnotes 2-chloroethyl vinyl ether with the following: "Due to instability of 2-chloroethyl vinyl ether, we cannot guarantee the concentration of this component." These problems with 2-chloroethyl vinyl ether impact the ability of SL to consistently analyze for this compound within the method requirements or PQL. If the requirements or PQL cannot be met for 2-chloroethyl vinyl ether, the appropriate flag should accompany the data for this compound in the report.

Semivolatiles/Pesticides/Herbicides

Initial calibration is performed upon instrument startup and whenever a CCC standard fails the acceptance criteria. A five-point standard curve is prepared using all target compounds. The low standard concentration is near but above the MDL and the high standard defines the usable linear range of the detector.

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After the five standards are injected, the computer software generates a calibration curve using either internal standard or external standard methodology. The analyst chooses the best fit type for each compound, either linear or quadratic. The analyst should inspect the curves before proceeding with sample analysis. An alternative to quantitation from a calibration curve is quantitation from an average response factor as long as the minimum XRSD criterion is met. The XRSD criteria are as follows:

- < 10% for 600-series methods.
- < 20% for 8000-series methods.
- 3. \leq 20% for 500-series methods, except Method 504 must be < 20%.

CCC standards are analyzed at the intervals specified in the methods. The 8000-series methods specify a CCC standard at the beginning of each working day and after every 10 samples thereafter if needed for further sample analyses. The 600-series methods specify a CCC standard at the beginning of each working day. The 8000- and 600-series methods CCC standard acceptance criteria are \pm 15% difference from the true value. The 500-series methods specify a CCC standard at the beginning of each work day. An additional CCC standard, different in concentration from the initial standard, must be run at the end of the work day when using the external standard calibration technique for methods 507, 508, and 515.1. The acceptance criteria for these CCC standards is \pm 20% difference from the true value. The 500-series methods allow a single point calibration as an alternative as long as the response produced by an unknown in the sample extract is \pm 20% of the standard response.

If the CCC standard fails acceptance criteria, another CCC standard may be analyzed. If the second standard also fails, the initial calibration must be repeated.

The above calibration procedures meet or exceed EPA method requirements.

The CLP protocol differs from the other EPA methodologies. Calibration curves with a minimum of three points are kept on record at the lab. The CLP statements of work for 2/88 and 3/90 (OLMO1.6) are followed as written.

9.4.4 GC/Mass Spectrometer

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for semivolatile GC/MS tuning with DFTPP and volatile tuning with BFB are given below. Mass calibration is performed as an integral part of tuning. Tuning is performed at the beginning of each 12-hour clock for each GC/MS in accordance with EPA methods.

SEMIVOLA	SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION (DFTPP)						
m/e	Ion Abundance Criteria						
51	30-60% of mass 198						
68	< 2% of mass 69						
70	< 2% of mass 69						
127	40-60% of mass 198						
197	< 1% of mass 198						
198	Base peak, 100% relative abundance						
199	5-9% of mass 198						
275	10-30% of mass 198						
365	> 1% of mass 198						
441	Present but less than mass 443						
442	> 40% of mass 198						
443	17-23% of mass 442						

	VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION BROMOFLUOROBENZENE (BFB)							
m/e	Ion Abundance Criteria							
50	15.0 - 40.0% of mass 95							
75	30.0 - 60.0% of mass 95							
95	Base peak, 100% relative abundance							
96	5.0 - 9.0% of mass 95							
173	Less than 2.0% of mass 174							
174	Greater than 50.0% of mass 95							
175	5.0 - 9.0% of mass 174							
176	Greater than 95.0 %, but less than 101.0% of mass 174							
177	5.0 - 9.0% of mass 176							

Initial calibration is performed at instrument startup and whenever a CCC standard fails acceptance criteria. A five-point standard curve is prepared containing all target compounds. Concentrations are those defined by CLP, which are also appropriate for other EPA methodology.

Response factors are generated for each compound. The acceptance criteria used to assess the calibration are those specified in SW-846 for the 600-and 8000-series methods and in the various CLP SOWs for CLP analyses. These are as follows:

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Semivolatiles								
	Initial Calibration	Continuing Calibration Check						
625 and 8270	< 301 RSD for CCCs	≤ 30% difference for CCCs						
Semivolatile CLP 2/88 SOW	< 30% RSD for CCCs	≤ 25% difference for CCCs						
625, 8270, CLP 2/88 SOW	≥ 0.050 SPCCs	≥ 0.050 for SPCCs						
CLP 3/90 SOH	As specified in 6/91 Revision of	Hethod (OLM01.6)						
525	S 30% RSD or alternatively generate linear, 2nd order, or 3rd order calibration curve	\$ 30% difference or alternatively (using analyst discretion), all analytes fall on the curve from the initial calibration						

	Volatiles	
	Initial Calibration	Continuing Calibration Check
624	< 30% RSD for CCCs	20 ug/L standard meets limits specified in Q Table
8240 + CLP 2/88 SOW	< 30% RSD for CCCs	≤ 25% difference for CCCs
624, 8240, CLP 2/88 SOH	≥ 0.300 for SPCCs (except Bromoform ≥ 0.250)	≥ 0.300 for SPCCs (except Bromoform ≥ 0.250)
CLP 3/90 SOW	As specified in 6/91 Revision	of Method (OLMO1.6)
524.2	≤ 20% RSD or alternatively generate linear, 2nd or 3rd order curve	± 30% difference or alternatively) using analyst discretion), all analytes must fall on the curve from the initial calibration

CCC standards are analyzed at the intervals specified in the methods. These intervals are as follows:

- 1. 500-series -- every 8 hours
- 2. 600-series -- every working day
- CLP & 8000-series -- every 12 hours.

If the CCC standard fails acceptance criteria, another CCC standard may be analyzed. If the second standard also fails, the initial calibration must be repeated.

Sample quantitation is based on the average RF or curve (when RTE data systems are not available) from the initial calibration for 500-, 600-, and 8000-series methods and the single point RF from the continuing calibration standard for CLP.

Hexachlorophene exhibits very poor chromatographic behavior within the limits of the working calibration range. If this compound is not detected, ND (not detected) will be reported rather than a detection limit.

2-Chloroethyl vinyl ether exhibits erratic chromatographic behavior. The Supelco, Inc. Purgeable A Mixture footnotes 2-chloroethyl vinyl ether with the following: "Due to instability of 2-chloroethyl vinyl ether, we cannot guarantee the concentration of this component." These problems with 2-chloroethyl vinyl ether impact the ability of SL to consistently

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analyze for this compound within the method requirements or PQL. If the requirements or PQL cannot be met for 2-chloroethyl vinyl ether, the appropriate flag should accompany the data for this compound in the report.

9.4.5 High Performance Liquid Chromatographs

Initial calibration is performed at instrument startup, following instrument maintenance or change in conditions, and whenever CCC fails acceptance criteria.

A three-point curve is prepared for 500- and 600-series methods. Five points are used for 8000-series methods. The low standard is near the PQL and the high standard defines the usable linear range of the detector.

After the three- or five-point standards are analyzed, response factors are generated by the data systems or manually. Due to limited data system capabilities, RSD criteria of 10% for the 600-series methods and 20% for the 500- and 8000-series methods are applied. If the maximum RSD criteria are met, the average RF is used for quantitation.

A CCC using a mid-level standard is performed at the beginning of each working day and after every ten samples. Acceptance criteria are less than or equal to 10% difference from the average RF for the 600-series methods, \leq 20% D for the 500-series methods, and \leq 15% D for the 8000-series methods.

If the CCC standard fails acceptance criteria, another CCC standard may be analyzed. If the second standard also fails, the initial calibration must be repeated.

9.5 Field Instrument Calibration

Calibration of field instrumentation (conductivity meters, pH meters, DO meters, and turbidimeters) is performed in the field prior to use, in accordance with the DER Calibration and Use of Field Meter SOP, revised Oct. 18, 1991. All calibration data are documented in a bound field notebook.

9.6 Calibration Documentation

All calibration records including raw data, response factors, standard concentrations, curves, reduced data, and instrument settings or conditions are stored and archived according to laboratory standard operating procedures. Current chromatograms, curves, and results transcribed onto forms are kept at the analysts' workstations and periodically archived into a data storage area. Initial and continuing calibrations are stored by date for ease of location. All standard ID numbers appear on graphs, plots, chromatograms, or curves for traceability purposes.

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10.0 PREVENTIVE MAINTENANCE

10.1 Maintenance Schedule

All Savannah Laboratories facilities are equipped with up-to-date computerized instrumentation. In order to gain maximum performance and minimize downtime, regular inspection, maintenance, cleaning, and servicing of all laboratory and field equipment is performed according to the manufacturers' recommendations. A maintenance log is kept for each piece of laboratory and field instrumentation, detailing any malfunction and the steps taken to correct the problem. Routine repairs and maintenance are performed and documented by the analyst responsible for the particular instrument. Non-routine maintenance is signed and dated by the analyst or repair technician. Routine maintenance procedures for laboratory instrumentation are given in Table 10.1. The frequencies of routine maintenance procedures for Savannah Laboratories' field instrumentation are given in Table 10.2.

Maintenance contracts are carried for most instrumentation, and close contact is maintained with service personnel to provide optimum instrument functioning.

An extensive spare parts inventory is maintained for routine repairs at the facilities, consisting of GC detectors, AA lamps, fuses, printer heads, flow cells, tubing, certain circuit boards and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be exchanged among the labs.

Equipment such as refrigerators, ovens, and incubators are not calibrated per se, but are periodically checked with calibrated thermometers. Refrigerators and incubators are checked twice daily and the temperatures documented in a notebook. Sample storage refrigerators must be $4 \pm 2^{\circ}$ C. All thermometers are calibrated annually against an NIST-certified thermometer.

Electronic analytical balances are calibrated daily with internal mechanisms if available. Calibration checks are performed and documented on all balances at least weekly with Class S weights and must meet the criteria given in Table 10.3.

10.2 Contingency Plan

In general, each facility has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation within each facility, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Savannah Laboratories location (where identical SOPs, QA procedures and instrument are utilized). When shipping samples to another facility, interdivisional chain-of-custody procedures are followed as given in Section 7.

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LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
	Se	rvic	e Ir	terv	7al	_		
EQUIPMENT ITEM	D	W	М	Q	A	SERVICE LEVEL		
ICAP								
Profile	х					Profile on a daily basis.		
Nebulizer	х					Inspect and clean. Replace tubing daily. Check flow rate.		
Filters		х				Inspect and clean.		
Spray Chamber			х			Inspect and clean.		
Quartz Torch	•		x			Clean and realign.		
D-Shaped Mirrors				X		Inspect mirror surface and replace if necessary.		
SMITH-HIEFTJE FURRACE AA SPEC	TROPHOTO	HETER						

CONDUCTABLE METER

Filters		х			Inspect and clean.
Spray Chamber			X		Inspect and clean.
Quartz Torch			x		Clean and realign.
D-Shaped Mirrors				X	Inspect mirror surface and replace if necessary.
SMITH-HIEFTJE FURNACE AA SPECTR	OPHOTO	HETER			
Sapphire Window	х				Remove and clean with N-Propanol.
Flow Rate	х				Place 10 mL DI water in a 10-mL cylinder. Push Neb. Air button and run one minute. Flow should be 2.0 to 2.5 mL.
Graphite Tube	х				Replace if necessary and condition before use.
Quartz Windows	х				Clean window with lint-free cloth and distilled water.
Contact Rings and Plates				x	Replace contact rings if they are worn.
Filters		х			Remove filter from instrument, clean with water and mild soap.
ZEEMAN FURNACE AA SPECIROPHOTO	ETER				
Check sampler syringe for air	х				Flush syringe if necessary.
Graphite Tubes	х				Replace if necessary and condition before use.
Graphite Electrodes				х	Replace contact rings if they are worn.
Quartz Windows	х				Remove and clean with lint-free cloth and DI

Graphice Liectiones				^_	Replace contact lings if they are worn.
Quartz Windows	x				Remove and clean with lint-free cloth and DI water and/or alcohol.
CONTINUEM FURNACE AA SPECTRO	PHOTOMETE	R			
Quartz Windows	х				Remove and clean with lint-free cloth and DI water.
Graphite Tubes	Х				Replace if necessary and condition before use.
Contact Rings and Plates				x	Replace contact rings if they are worn.
Filters		х			Remove filter from instrument, clean with water and mild soap.
D2 Arc Lamp				x	Check lamp. Adjust or replace as necessary.
TURBIDIMETER			X	-	Focus optics.

Inspect and replatinize cell as necessary.

TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Se	ral				
EQUIPMENT ITEM	D	D W H Q A		A	SERVICE LEVEL	
pë Meter	х					Inspect probe membrane, filling solution level.
DRYING OVEN	х				_	Verify correct temperature with calibrated thermometer.
ANALYTICAL BALANCE		х				Check calibration with class S standard metric weights. Annual inspection.
TOP LOADER BALANCE		х				Check calibration with class S standard metric weights. Annual inspection.
ION CERCHATOGRAPE						
AS3 Column				х		Inspect quarterly or as required.
AS3 Guard Column				х		Inspect quarterly or as required.
Pump Pistons					х	Inspect annually.
AUTOANALYZER						
Pump Platen		X				Inspect weekly and replace as required.
Pump Tubes	x					Inspect and replace as needed.
Flow Cell		Х				Inspect and clean.
BLOCK DIGESTOR				Х		Check calibration against thermometer.
UV/VIS SPECTROPHOTOMETER					х	Semiannual check for wavelength verification.
IR SPECIROPHOTOMETER		X				Inspect and clean exposed optics weekly, if necessary.
ION SELECTIVE ELECTRODE			Х			Inspect and polish electrode.
BOMB CALORIMETER		x				Inspect seals, replace if necessary.
DISSOLVED OXYGEN METER	х					Check probe membrane for deterioration. Replace as necessary.
BOD INCUBATOR	х					Temperature checked twice daily.
BACTERIOLOGICAL INCUBATOR	x		<u> </u>			Temperature checked twice daily.
AUTOCLAVE		х				Seals inspected and replaced as necessary.
WATERBATE .	Х					Temperature checked twice daily.
TCLP EQUIPMENT				х		Check rotation rate quarterly.
GAS CERCHATOGRAPE - SEMIVOLAT	ILES					
Autosampler System	x					Check daily for correct operation. Syringe and tubing solvent cleaned daily. Needles and tubing replaced as needed.
Septa	x					Replace autosampler septa daily and injector as needed.
GC Columns (Packed)		X				Change glass wool plugs at front of column.
GC Capillary Columns	Х					Inspect daily. Change glass sleeve insert as needed and cut front of column if necessary.

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TABLE 10.1

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Se	rvic	е Тп	terv	ra l	
EQUIPMENT ITEM	D	W	м	Q	A	SERVICE LEVEL
ECD				-	х	Semiannually cleaned and leak tested by service technician.
FID					x	In-house cleaning as needed,
Carrier Gases		х				Tanks are changed when pressure reads 500 to ensure purity.
Oxygen Trap				х		Inspect and replace as necessary.
GAS CHROMATOGRAPH - VOC						
Column	х					Checked daily. Repack glass wool and replace column as needed.
Septum	Х					Checked daily. Replace as necessary.
Gas Tank	х					Levels checked daily. Replace when pressure < 500 psi.
Oxygen/Moisture Trap				X		Inspect and replace as necessary.
Particulate Trap					x	Checked and replaced if problem in GC flow rate.
Hall Detector	х		-			Checked daily for proper operation and response.
FID	Х					Checked daily for proper operation and response.
PID	Х					Checked daily for proper operation and response.
GC/MS						
Column	x					Front portion of column checked/maintained daily for contamination; replace every 1 month or as needed.
Septum	х					Changed daily.
Injection Port Liner	Х	L. <u>.</u>				Changed daily.
Splitless Disc	X					Changed daily.
Autosampler	X					Checked daily for proper function.
Rough Pump				x		Oil changed to ensure proper operation.
Turbo Pump				x		Turbo molecular pump oiled as needed by instrument service representative.
Mass Spectrometer				X		Cleaning of source every 1 month or as needed.
Tape Head					X	Cleaned after each tape.
Tape Drive					x	Cleaned annually.
PURGE AND TRAP						
Sorbent Trap	х					Checked daily. Replace and condition as necessary.
Purge Flow	х					Checked daily, adjust as needed.
Gas Tank	X					Check daily.

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TAROPATORY	ECHTOMENT	PREVENITVE	MAINTENANCE	SCHEDIILE
THIDOMETONE		T V T 1 T T Y T 1 T	**************************************	

	Se	rvic	e In	terv	al	
EQUIPMENT ITEM	D	W	М	Q	A	SERVICE LEVEL
TOC ANALYZER						
Pump Tubes		х				Inspect and replace if necessary.
Flow rate			X			Check and adjust if necessary.
Detector Windows					х	Check and clean if necessary.
TOX ARALYZER						
Pyrolysis Tube		х				Inspect and clean or replace if necessary.
Electrodes		х				Inspect and clean if necessary.
Electrolyte	х					Replace as necessary.
HPLC SYSTEMS						
Pumps	x					Filter all solvents, water, and extracts if pressure buildup occurs. Visual leak check. Prime pumps at startup.
Pumps				Х		Inspect seals, replace as needed.
Columns	х					Check for pressure buildup; store with ends capped in appropriate mobile phase. Visual leak check.
Detector fittings	х					Visual leak check.
Detector optics	Х					Inspect removable filters for dust, fingerprints. Clean as needed.
Detector optics					х	Replace lamps as needed.
Autosampler	х					Checked daily for proper operation. Clean, lubricate moving parts as needed.
Gases for sparging and autosampler operation		х				Change tanks when pressure reads 500 ps:.
TENNELEC LB5100						
Sample changer				х		Inspect moving parts, lubricate as needed.
Detector	х					Checked daily for proper operation and response. Serviced by manufacturer only.
Detector gas			x			Change tank when pressure reads 500 psi. Allow new tanks to dissipate radon for two weeks before use.
Flow meter	Х					Checked daily for proper operation.

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TABLE 10.2

FIELD EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

	Se	Service Interval				
EQUIPMENT ITEM	D	W	М	Q	A	SERVICE LEVEL
TURBIDIMETER HACH 16800/DRI- 15C	x					Inspect and replace cell as needed.
CONDUCTANCE METER YSI 33				х		Inspect and replatinize cell as necessary.
FISHER AND CRION PH METERS	x					Inspect probe membrane, filling solution level.
YSI MODEL, 50B/51B DISSOLVED OXYGEN METER	x					Check probe membrane for deterioration. Replace as necessary.
CORNING CHECKMATE 90 pH/SC/DO/T METER	х					Check probe, call, membrane.
FISHER/SLOPE WATER LEVEL METERS	x					Check probe cord for integrity/cleanliness, meter for response.

TABLE 10.3		
BALANCE CALIBRATION CHECKS		
Analytical Balance		
Class S Weight	Tolerance	
0.01 g	± 0.0002 g	
0.1 g	± 0.0002 g	
0.5 g	± 0.0004 g	
1 g	± 0.0004 g	
10 g	± 0.0005 g	
50 g	± 0.0010 g	
Top-Loading Balance		
Class S Weight	Tolerance	
0.1 g	± 0.02 g	
0.5 g	± 0.02 g	
1 g	± 0.04 g	
5 g	± 0.04 g	
10 g	± 0.05 g	
50 g	± 0.10 g	

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cleaning, contaminated blank source water, sample contamination during storage and transportation due to exposure to volatile organics (e.g., gasoline fumes) and other environmental conditions during the sampling event.

PREPARATION: Trip blanks are prepared prior to the sampling event either by the laboratory providing sample containers, or by field team personnel who are responsible for the initial preparation of sample containers and field equipment. The water must be free of volatile organic contaminants. Any appropriate preservatives must be added at the time that the blanks are prepared. The sample containers are sealed, labeled appropriately, and transported to the field in the same sampling kits as the sample vials. These blanks are not to be opened in the field. They are to be transferred to the sample container designated for volatile sample storage and transport and accompany the samples to the laboratory. Subsequent blanks (field and equipment) for volatile organics should use the same source water as the trip blanks, unless the water used for field and equipment blanks can be proven equivalent.

FREQUENCY: One trip blank for each volatile organic analysis (601, 602, 624, etc.) shall be provided per cooler used for storing and transporting volatile sample vials. If a laboratory requires submission of multiple vials for a method, the same number of vials must be submitted for the trip blank.

11.1.2 Field Blanks

PURPOSE: Field blanks are used to evaluate the effects of on-site environmental contaminants, the purity of reagents used as preservatives or additives and the general sample container filling/collection techniques. Field blanks are recommended for all parameters.

PREPARATION: Field blanks are prepared <u>on-site</u> by filling the sample container(s) with analyte-free water, adding preservatives, sealing the containers and completing the appropriate documentation. The field blanks must be handled in the same manner as the sample group for which it was intended (i.e., blanks must be stored and transported with the sample group).

NOTE: The water for VOA field blanks should be equivalent to the trip blank water (see Trip Blank Preparation).

FREQUENCY: One field blank per parameter group per day or at a frequency of 5% of the samples in the parameter group per day, whichever is greater.

11.1.3 Equipment Blanks

PURPOSE: Equipment blanks are required if sampling equipment is precleaned or field-cleaned. These blanks are used to determine the effectiveness of field cleaning procedures as well as to reveal those sources of contamination that may be found in a trip blank. Equipment

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blanks are recommended for all parameter groups and matrices to be collected and analyzed.

PROCEDURE: The final rinse water (analyte-free) shall be rinsed on or through the sampling equipment, whether precleaned or field cleaned, collected, and placed in appropriate preserved containers. These blanks must be stored and transported with the samples.

NOTE: The water used for volatile equipment blanks should be from the same or equivalent source as the trip blank water.

FREQUENCY: When less than five samples of a similar matrix are taken, one equipment blank prepared on-site for precleaned or field-cleaned equipment must be collected and analyzed for each parameter.

When five to ten samples of a similar matrix are taken, one equipment blank must be collected on field-cleaned equipment or one on-site blank must be collected in precleaned equipment if no equipment is cleaned in the field.

For sampling events involving ten or more samples, a minimum of one blank must be taken on precleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices. One blank must be taken on field-cleaned equipment or at the rate of 5% (whichever is greater) of the samples in each analyte group for all matrices.

11.1.4 Field Duplicates

Field duplicates are taken, analyzed, reported and invoiced when requested by the client or specified by a project specific QA plan. Savannah Laboratories recommends that a minimum of one duplicate or 10% of samples be taken for all parameter groups and matrices to be collected and analyzed.

11.1.5 Field QC Summary

The frequency of field blanks and duplicates is summarized below:

No. Samples	Precleaned Equipment Blanks	Field-cleaned Equipment Blanks	Trip Blank (VOCs)	Duplicates
10+	Minimum of one then 5%	Minimum of one, then 5%	One per cooler	Minimum of one then 10%
5-9	One*	One*	Not required	One
< 5	One*	One*	Not required	Not required

^{*} Note: For nine or fewer samples, one equipment blank is required from either precleaned or field-cleaned equipment.

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If any equipment is cleaned in the field, the blank is to be taken from the field-cleaned equipment.

11.2 Laboratory QC Checks

The laboratories employ control samples to assess the validity of the analytical results. Determination of the validity of sample results is based on the acceptance criteria being met by the control sample. The acceptance criteria for each type of control sample are defined in the appropriate SOP. These acceptance criteria are determined from historical data, and meet the EPA CLP acceptance criteria as a minimum. The control samples are analyzed in the same manner as the field samples. QC check samples are analyzed on an analytical batch frequency unless otherwise stated. An analytical batch is defined as a group of samples which are processed as a unit. If the number of samples in the group is greater than 20, each group of 20 samples or less is handled as a separate batch.

Other QC check samples are analyzed for performance evaluations or as part of internal or external audits as given in Section 14. Blind QC check samples are analyzed at a minimum in duplicate and at least semiannually. Results of any unacceptable QC check sample results obtained during DER-reportable project analysis are submitted to DER QAS in the project report as discussed in Section 15.

If particular laboratory method QC requirements are more stringent than the general procedures given below, the method QC requirements are followed.

11.2.1 Organics

Method Blanks: A method blank will be run for each batch of samples. A blank is a clean sample (containing no reportable analyte).

Lab Control Standards: Blank spikes or lab control standards will be run with each batch of samples processed.

Surrogates: Appropriate surrogates (see Tables 5.1 and 5.2) will be added to all samples, standards and blanks.

Matrix Spikes: Matrix spikes will be run with each batch at a frequency of 5% of samples. The CLP matrix spiking compounds will be used for all GC/MS semivolatile, volatile and chlorinated pesticides/PCB analyses (by GC). Appropriate matrix spikes will be used for other chromatographic methods.

Matrix Spike Duplicates/Sample Duplicates: Duplicate samples or matrix spikes will be run with each batch or at a frequency of 5% of samples. In cases where duplicate matrix spikes are used, precision data are obtained on only the matrix spiking compounds.

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11.2.2 Inorganic and General Chemistry

Calibration Blanks: Calibration blanks are nondigested blanks which are run at a frequency of 10% of samples.

Method Blanks: Method blanks should be run with each batch at a frequency of 5% of samples of the same matrix.

Lab Control Standards: Blank spikes or lab control standards will be run with each batch of samples processed.

Matrix spikes: Matrix spikes will be run at a frequency of 5% of samples.

Duplicates: Duplicate samples or duplicate matrix spikes will be run at a frequency of 5% of samples.

In cases where batch QC is sufficient, the matrix spike/duplicate will be on a field replicate (if available) or a laboratory provided sample of the same matrix. In the case of nonliquid inorganics, the digestion blank or extraction blank will be spiked.

Cost for analyses of batch QC matrix spikes/duplicates, laboratory blanks and blank spikes (lab control standards) are included in the individual costs for analysis.

For CLP protocols or other cases where "sample specific" (non-batch) QC is required, matrix spike/duplicates will be conducted on replicate samples provided by the client. In this case, matrix spikes/duplicates analysis will be invoiced as samples. If the client does not provide sufficient sample replicates for matrix spikes/duplicates, laboratory generated samples will be provided.

11.2.3 Microbiology

Quality control checks are routinely performed for all microbiological analyses. Strict requirements for the house deionized water must be met before it can be used in any testing. Each monitored parameter, its monitoring frequency, and its acceptance limits is as follows: residual chlorine, monthly, <0.1 mg/L; trace metals (total Cd, Cr, Cu, Ni, Pb, Zn), annually, < 1.0 mg/L; conductivity, daily < 1.0 umho/cm; heterotrophic plate count, monthly, < 1000 CFU/mL; and suitability (inhibiting residue), annually or for each new lot of detergent, ratio between 0.8 and 3.0.

Other laboratory QC practices are utilized to provide accurate microbiological results. These include the use of autoclave tape to insure proper sterilization of sample containers, media, etc. Incubators are maintained at $35 \pm 0.5^{\circ}$ C and water baths at $44.5 \pm 0.2^{\circ}$ C. Thermometers used for these monitoring purposes are calibrated annually against an NIST-certified thermometer. Other equipment, such as the dissecting microscope and colony counter are maintained in clean operating condition at all times.

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Microbiological samples are analyzed in duplicate at a rate of 10% of positive samples. Positive controls are analyzed in association with ONPG-MUG analyses for total coliform monthly or upon receipt of a new lot of reagents.

Blanks are routinely analyzed with microbiological samples. For membrane filter analyses, a sterile dilution water blank is run initially, after every 10 samples, and at the end of each analytical run. For MPN analysis, sterile dilution blank is added to a lauryl tryptose broth tube for a blank for each analytical run.

11.3 Routine Method Used to Assess Precision and Accuracy

Control charts (Figures 11.1 and 11.2) for precision and accuracy are setup for each parameter immediately after the method is validated. Control charts are based on procedures in The Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA, 1979) and contain both "Warning Limits" (± 2 standard deviations) and "Control Limits" (± 3 standard deviations). The initial limits used for a parameter are the values obtained from the method validation procedure (40 CFR Part 136). Control limits are updated annually for all parameters. A minimum of ten data points is used to update these limits. Formulas used for calculations of precision and accuracy are provided in Section 5.0.

Accuracy and precision limits are established for a specified concentration range. Concentration is divided into three ranges: low, mid, and high. Low level is defined as concentrations from the minimum detection limit to a level five times the MDL. Mid level is defined as the mean level between the minimum detection level and the upper and of the linear range. High level is defined as the concentration at the upper end of the linear range. Further information on these ranges is found in Section 9. The procedures used to determine the precision and accuracy targets in Section 5 are given in Table 11.1.

11.4 Method Detection Limits and Reporting Limits

Method detection limits (MDLs) are determined annually in accordance with the procedures in SW-846 and Appendix B of 40 CFR Part 136. This procedure includes analyzing seven prepared spikes or standards in reagent water at levels 3-5 times the estimated detection limit. The standard deviation of the seven replicate measurements is calculated, and the MDL is computed by multiplying this standard deviation by 3.14 (the Student's t value appropriate for a 99% confidence level with seven replicates).

The method detection limit (MDL) calculated by the procedure described above is defined as the minimum concentration of a substance that can be measured in reagent water and reported with confidence that the analytical concentration is greater than zero.

For other protocols (i.e., Contract Laboratory), other procedures are used to estimate detection limits.

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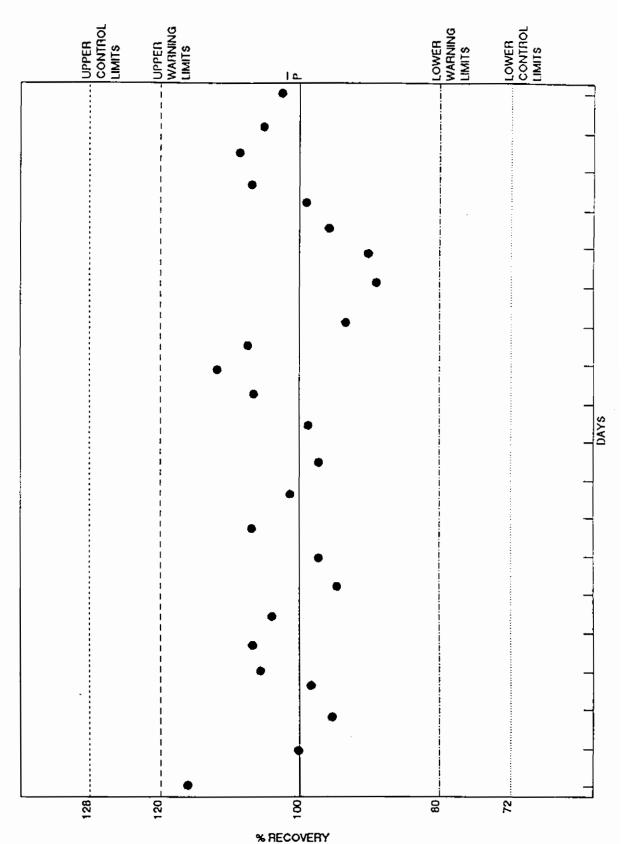
Since MDLs are based on the analyses of standards in reagent water, they are not useful in reporting data for most environmental samples. Thus, practical quantitation limits (PQLs) are used for reporting a non-detected parameter. PQLs are defined as the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

The new term from SW-846, Estimated Quantitation Limits (EQL) is used interchangeably with PQL. In all cases, PQLs are greater than MDLs. When PQLs are defined in SW-846 or the CLP protocols (CRDLs), these defined PQLs are generally used in data reporting provided they are achievable and within the range of 10 times the standard deviation used in determining the MDL and 10 times the MDL.

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FIGURE 11.1

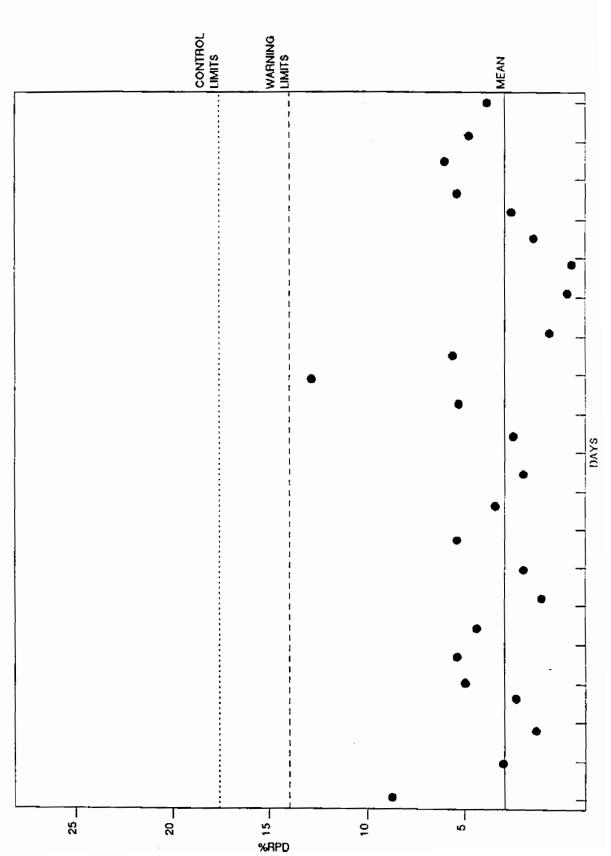
EXAMPLE OF CONTROL CHART FOR % RECOVERY



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FIGURE 11.2

EXAMPLE OF CONTROL CHART FOR % RPD



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Methods used to Generate Accuracy and Precision Targets					
Method	Purpose	Concentration Level	Method References		
Quality Control Check Standards (QCCS)	Accuracy	Mid Level	All metal, general, and organic methods for which a QCCS is available.		
Quality Control Check Standards (QCCS)	Precision	Mid Level	All metal, general, and organic methods for which a QCCS is available.		
Duplicate Samples	Precision	Mid Level	All methods for which a QCCS is not available		
Matrix Spikes	Accuracy	Mid Level	Methods for project- or agency-specific requirements.		
Matrix Spike Duplicates	Precision	Mid Level	Methods for project- or agency-specific requirements.		

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12.0 DATA REDUCTION, REVIEW, AND REPORTING

12.1 Introduction

In order to provide the highest quality data possible, an extensive system for data reduction, review, and reporting has been implemented.

12.2 Sample Custody

Upon receipt of the samples, the custody forms are checked against the sample identifications listed on the containers by the sample custodians, and a unique SL log number is assigned to each sample group. Any discrepancies are noted, including cooler temperatures, broken bottles and/or misidentified samples. The data manager or the project manager then notifies the client if discrepancies exist.

After receipt, the samples are delivered to the appropriate laboratory sections where the samples are checked for proper preservation and this information is recorded in bound notebooks when applicable. When necessary, the samples are then stored in refrigerators that are monitored twice daily for temperature.

12.3 Organization and Initiation of Sample Analyses

The key to Savannah Laboratories' sample flow, analysis, data and QA review and archiving, and reporting system is the single LIMS network which controls the day to day production of the laboratories. This system, which is summarized in Figure 12.1, provides project managers, QA personnel, and all analysts immediate information on the status of any sample in all five facilities. This system schedules and prioritizes all work, provides a mechanism for sample tracking, review of reportables and QC data, generation of reports and invoices, and archiving of all reports and associated QC data.

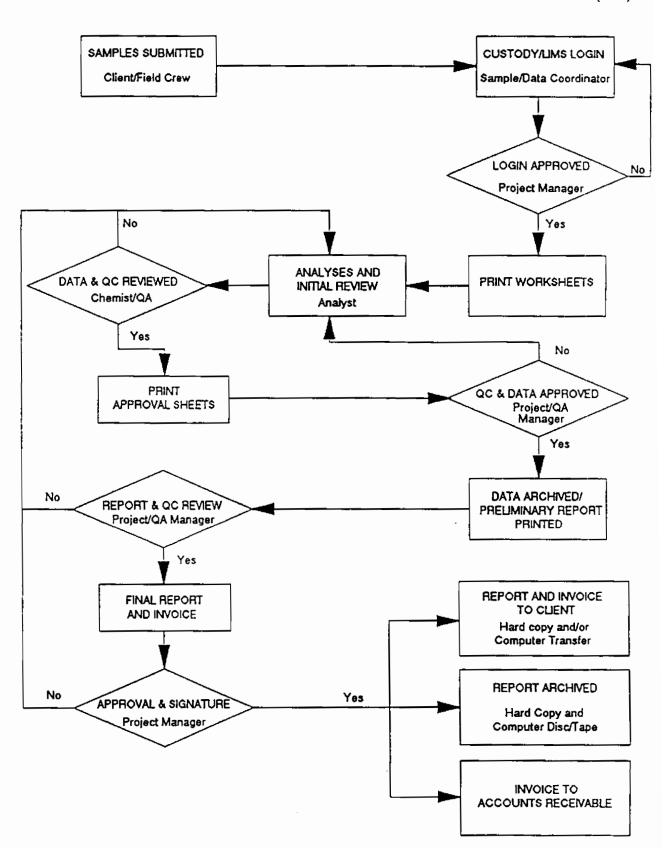
Upon receipt of custody forms, the project manager instructs data management personnel to log the sample analysis request and identification into the LIMS. The LIMS is based on an ADDS Mentor 7000 computer (NCR) which links the Tallahassee, Mobile, Deerfield Beach, Tampa, and Savannah facilities via telephone multiplex. This enables any project manager, section manager, QA manager, laboratory director, or chemist with authority to access projects to check the status of a project.

If special handling or data packaging is required, the QA department receives copies of the custody forms and computer acknowledgement and then initiates a QA project file and determines the sample batching. A sample delivery group (SDG) sheet is established and distributed to all affected departments including the various laboratory chemists, project managers, and section managers.

After the sample analysis request is logged into the LIMS and approved, the LIMS generates worksheets which are printed and distributed three times weekly.

Figure 12.1

FLOW CHART OF SL COMPUTERIZED LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)



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12.4 Sample Analysis and Data Reduction

Through the use of the worksheets and/or SDG sheets, the samples are prepared following the procedures given in each of EPA's approved methods. The preparation information is recorded in bound notebooks throughout the laboratory.

12.4.1 Data Reduction

Most sample concentration results are read directly from instrumentation without further reduction or calculations. Dilution factors are applied upon the dilution of samples having concentrations above the calibration range. In many cases, these are input into the instrument computer and correct results are calculated automatically. In other cases, a manual calculation may be made. All soil/solid waste concentration results for all laboratory sections must be calculated on a dry weight basis prior to reporting by dividing the instrument result by the dry weight fraction.

Other than the cases discussed above, data obtained by the following method/instrument are directly reportable: volatile GC, volatile GC/MS, semivolatile GC/MS, metals ICP, metals AA, general chemistry automated colorimetry, TOC, DO, turbidity, and pH.

Methods data requiring reduction prior to reporting include semivolatile GC, titrimetric methods, BOD, COD, conductivity, manual UV/VIS/IR, residue, and TOX.

Table 12.1 gives equations used in computer-controlled instrumentation for data reduction as well as equations used for the manual calculation of reportable concentration results.

All laboratory pH meters are temperature compensated. Laboratory conductivity is always measured at 25°C.

The laboratory raw data containing the instrument-generated reports, manually calculated results, and all supporting preparation, calibration, and analytical data are retained at the individual work stations until reports are issued unless additional handling or data packaging is required.

All field pH and conductivity meters are temperature compensated. Cell constants for field conductivity meters are determined by laboratory personnel annually as given in Section 9.4.2. Field conductivity is calculated as given in Table 12.1. All other field data are read directly from instrumentation.

Bound field notebooks are used for documentation of required data reduction. Calculations are recorded in waterproof ink.

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

	Reporting Units		
N/A Extractables by GC/MS [Internal Standard Method (625 and 8270)]	Water	Solid*	
Response Factor = As X Cis (RF) Ais X Cs	ug/L (or mg/L)	ug/kg (or mg/kg)	
As = area of the characteristic ion of standard Ais = area of the characteristic ion of internal standard Cs = concentration of standard (ug/L) Cis = concentration of the internal standard (ug/L)			
Water = As x Cis Conc., ug/L Ais RF			
As = area of the characteristic ion (sample) Ais = area of the characteristic ion (internal standard) Cis = concentration of the standard (ug/L) RF = response factor			
Sediment = ug of internal standard x As x 1 Conc., ug/kg (kg of sample)(X solids x .01) Ais RF			
As = area of the characteristic ion (sample) Ais = area of the characteristic ion (internal standard) RF = response factor			
OC by GC/MS [Internal Standard Method - See section on BN/A]			
	ug/L (or mg/L)	ug/kg (or mg/kg)	
Cs by GC Response Factor = ug/L of compound to be measured			
Response Factor = ug/L of compound to be measured (RF) peak height Water = RF x peak height x dilution factor			
Response Factor = ug/L of compound to be measured (RF) peak height Water = RF x peak height x dilution factor Conc., ug/L Sediment = RF x peak height x liter equivalent of std. volume Conc., ug/L (kg of sample)(X solids x .01)			
Response Factor = ug/L of compound to be measured (RF) peak height Water = RF x peak height x dilution factor Conc., ug/L Sediment = RF x peak height x liter equivalent of std. volume Conc., ug/L (kg of sample)(X solids x .01)		(or mg/kg)	
Response Factor = ug/L of compound to be measured (RF) peak height Water = RF x peak height x dilution factor Conc., ug/L Sediment = RF x peak height x liter equivalent of std. volume Conc., ug/L (kg of sample)(X solids x .01) esticides/PCBs and Other GC Procedures Response Factor = ug of analyte (RF) (Standard) peak area Water Conc., ug/L = RF x peak area x extract volume in uL	(or mg/L)	(or mg/kg)	
(RF) peak height Water = RF x peak height x dilution factor Conc., ug/L Sediment = RF x peak height x liter equivalent of std. volume Conc., ug/L (kg of sample)(X solids x .01) esticides/PCBs and Other GC Procedures Response Factor = ug of analyte (RF) (Standard) peak area Water Conc., ug/L = RF x peak area x	(or mg/L)	(or mg/kg)	

TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporti	ng Units
Metals	Water	Solid*
Calibration curve construction	ug/L (or mg/L)	ug/kg (or mg/kg)
y = mx + b	}	
y = absorbance m = slope = <u>absorbance</u> concentration		
<pre>x = concentration (mg/L) b = y intercept</pre>		
Calculation of water sample concentration		
Water Conc., $ug/L = y - b \times dilution factor$		
Sediment Conc., mg/kg = mg/L x dilution factor x <u>final volume (liters) of digest</u> (kg of sample)(I solids x .01)		
UV/VIS and IR Procedures		
Calibration curve construction (see metals)	mg/L	mg/kg
Water Conc., mg/L = <u>y - b</u> x dilution factor m		
Sediment = mg/L x <u>liters of leachate (or digest)</u> Conc. (kg of sample)(X solids x .01)		
General Titrimetric Procedures		
Analyte, mg/L = N _{tytant} X Titer X eq. wt. X 1000 Vol. of sample titrated	mg/L	
BOD		
BOD, mg/L = (Int. DO - Final DO) - Seed Correction Factor Vol. fraction of sample	mg/L	
COD		
COD may! = (Blb tither a completion) V V V coop	mg/L	
COD, mg/L = (Blk titer - sample titer) X N, x X 8000 Vol. of sample, mL		

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TABLE 12.1

SUMMARY OF EQUATIONS USED IN CALCULATIONS

Equations	Reporti	ng Units
Conductivity	Water	Solid*
Cell constant = 1000 Observed conductivity of 1000 - μS/cm std.	μS/cm	
Residue		
Residue, mg/L = Total wt Wt. of dish or filter Vol. of sample, L	mg/L	
rox		
TOX, $\mu_B/L = (C1 + C2 - 2C3) \times \frac{1000 \text{ mL}}{\text{Vol. of sample}}$	mg/L	mg/kg
TOX, $mg/kg = \frac{instrument\ reading\ X}{\mu L\ injected} \times \frac{5}{dry\ wt.\ fraction}$		

^{*} Data for solid or semisolid sample are reported on a dry weight basis.

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12.4.2 Chromatographic and Data File Identification

Chromatograms and data files are given a unique alphanumeric identification by the chemists initiating the analyses in each section where appropriate. These file identification numbers reflect either the date the sequence was initiated (GC sections), the order in which the samples were analyzed (GC/MS sections), and/or the sample identification and log numbers given by the client and listed on the LIMS.

12.5 Data Transfer and Review

12.5.1 Data Transfer to LIMS

The analytical results are entered on the sectional worksheets after review. The worksheet data are entered into the LIMS by the data entry technicians.

After the data are entered into the LIMS, project manager approval sheets are printed and the project managers and one of the data managers check each worksheet against the information entered into the LIMS for transfer errors and anomalies.

12.5.2 Data Review

Laboratory analytical results are reviewed by the chemist responsible for the analysis and/or a peer chemist or a section supervisor. Prior to entering the reportable data into the LIMS, laboratory raw data have been reviewed, stamped, and signed to ensure that all of the method specifications have been met. This includes checking the extraction, digestion, distillation, and other preparation logs, as well as ensuring that all precision and accuracy requirements are addressed, and all steps of the analyses have been completed. If any problems arise during the analysis of the sample batch, it is the responsibility of the chemist and the section supervisor to bring this to the attention of the project manager, section manager, and QA manager through a written corrective action report.

The field/sampling manager is responsible for data review of all field-generated data. This includes verifying that all field descriptive data is recorded as per Section 6, that all field calibration requirements have been met as per Section 9, that all field QC data have met criteria given in Table 5.3, and that field data are entered accurately on worksheets.

For reports on which QA deliverables are required, data flags are used to inform the project manager and the client of any additional information that might aid in the interpretation of the data. The data flagging system incorporates the data qualifiers specified in the Contract Laboratory Program protocols, as well as additional flags used to help explain batch specific events.

When data acquisition and reporting have been completed, the project manager reviews and prepares the final report. Because the project managers have extensive experience in evaluating analytical data, they

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have developed both objective and subjective techniques for data review. Each value reported is reviewed in the context of the respective environmental matrix and all available QC/QA data. Outliers or other abnormal values are carefully scrutinized, and samples are reanalyzed if the abnormalities cannot be explained. Where there are cases in which the results from spiked samples suggest interferences, attempts are made to remove the interferences, or alternate analytical procedures are used. If the interference problem cannot be resolved, the data are flagged and/or a narrative is included with the report.

12.5.3 Special Project or Data Package Review

If special handling and/or data packages are requested by the client, the QA department also reviews the project report and the raw data. This includes checking to ensure that holding time requirements are met, reviewing internal chain of custody, recalculating results and detection limits, checking calibrations, reviewing all quality control data and/or control charts, and initiating any corrective action or reanalyses that might be appropriate.

If requested, the data packages are paginated, copied, and bound by the QA staff.

12.6 Reporting

The final report is printed and signed by the project manager after all review has been completed.

Figures 12.2 - 12.5 are examples of RESULTS ONLY SL Level 0, SL Level 1, SL Level 2, and SL Level 3 (CLP equivalent) typical reports for liquids samples. For CLP parameters, the CLP forms from the CLP SOW are generated by instrument software and are submitted to the client. If requested by the client or a project specific QA Plan, hybrid/custom reports or CLP data packages with diskette deliverables can be provided. All LIMS reports can be downloaded onto diskettes or most client's computers.

The data flags that may appear in a project report are defined on the signature page, and any additional comments are also footnoted on this page.

If data packaging is requested, a paginated, copied, and bound data package is provided in addition to the project report. The format of the project report and/or data package can be adjusted to meet the needs of the client.

12.7 Data Storage

The raw data are stored in metal filing cabinets at each work station until the cabinets are filled to capacity. The data are then transferred to a secured area and filed chronologically by laboratory section in banker's boxes for a period of 3-5 years. If the data are to be purged to the client or need to be separated from the general raw data files, the data can be boxed, labeled and stored in a separate secured area.

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Hard copies of all reports are maintained for 3-5 years in client file. All LIMS reports and associated QC data are kept for a minimum of three years on the LIMS hard discs or magnetic tape. All data on the LIMS are backed up daily on magnetic tape.

All in-lab data generated by computer systems are stored to tape when the capability exists. The tapes are labeled and stored at the individual work stations.

Keys to the data storage areas are retained by the QA staff and the section/department managers.

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FIGURE 12.2

EXAMPLE OF RESULTS ONLY REPORT

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LOG NO: SE-00010

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

Project: SL 0 (Results only) Report

Sampled By: Client

REPORT OF RESULTS

Page 1

	REPORT OF	RESULTS	Page 1
LOG NO SAMPLE DESC		LES	
00010-1 Water Sampl	e 1 (Collected 2-21-9		
PARAMETER		00010-1	
Purgeable Halocarbons (
Bromodichloromethane,		<1.0	
Bromoform, ug/l	_	<1.0	
Bromomethane, ug/1		<1.0	
Carbon Tetrachloride,	ug/l	<1.0	
Chlorobenzene, ug/l		<1.0	
Chloroethane, ug/l		<1.0	
2-Chloroethylvinyl Eth	er, ug/l	<1.0	,
Chloroform, ug/l		<1.0	
Chloromethane, ug/l		<1.0	
Dibromochloromethane,	ug/l	<1.0	
1,2-Dichlorobenzene, u	g/l	<1.0	
1,3-Dichlorobenzene, u	lg/1	<1.0	
1,4-Dichlorobenzene, u	g/l	<1.0	
Dichlorodifluoromethan	e, ug/l	<1.0	
1,1-Dichloroethane, ug	1/1	<1.0	
1,2-Dichloroethane, ug		<1.0	
1,1-Dichloroethene, ug		<1.0	
Trans-1,2-Dichloroethy		<1.0	
1,2-Dichloropropane, u	- '	<1.0	
Cis-1,3-Dichloroproper		<1.0	
Trans-1,3-Dichloroprop	-	<1.0	
Methylene Chloride, ug		<1.0	
1,1,2,2-Tetrachloroeth	nane, ug/l	<1.0	

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LOG NO: SE-00010

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

Project: SL 0 (Results only) Report

Sampled By: Client

REPORT OF RESULTS

Page 2

LOG NO SAMPLE DESCRIPTION , LIQUID SAMPLES		
00010-1 Water Sample 1 (Collected 2-21-91)		
PARAMETER	00010-1	*
Tetrachloroethene, ug/l	<1.0	
1,1,1-Trichloroethane, ug/l 1,1,2-Trichloroethane, ug/l	<1.0 <1.0	
Trichloroethene, ug/l Trichlorofluoromethane, ug/l	<1.0 <1.0	
Vinyl Chloride, ug/l Purgeable Aromatics (602/8020)	<1.0	
Benzene, ug/l Chlorobenzene, ug/l	<1.0 <1.0	
<pre>1,2-Dichlorobenzene, ug/l 1,3-Dichlorobenzene, ug/l</pre>	<1.0 <1.0	
1,4-Dichlorobenzene, ug/l Ethylbenzene, ug/l	<1.0 <1.0	
Toluene, ug/l Xylenes, ug/l	<1.0 <1.0	
Lead , ug/l	<5.0	

Methods: EPA 40 CFR Part 136

J. W. Andrews, Ph. D.

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FIGURE 12.3

EXAMPLE OF SL LEVEL I REPORT

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LOG NO: SE-00011

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

> Project: SL I Report Sampled By: Client

	REPORT OF RESULTS	Page 1
LOG NO SAMPLE DESCRIPTION ,	LIQUID SAMPLES	
00011-1 Water Sample 1 (Coll	ected 2-20-91)	
PARAMETER	00011-1	
Purgeable Halocarbons (601/8010)		
Bromodichloromethane, ug/l	<1.0	
Bromoform, ug/l	<1.0	
Bromomethane, ug/l	<1.0	
Carbon Tetrachloride, ug/l	<1.0	
Chlorobenzene, ug/l	<1.0	
Chloroethane, ug/l	<1.0	
2-Chloroethylvinyl Ether, ug/l	<1.0	
Chloroform, ug/l	<1.0	
Chloromethane, ug/l	<1.0	
Dibromochloromethane, ug/l	<1.0	
1,2-Dichlorobenzene, ug/l	<1.0	
1,3-Dichlorobenzene, ug/l	<1.0	
1,4-Dichlorobenzene, ug/l	<1.0	
Dichlorodifluoromethane, ug/l	<1.0	
1,1-Dichloroethane, ug/l	<1.0	
1,2-Dichloroethane, ug/l	<1.0	
1,1-Dichloroethene, ug/l	<1.0	
Trans-1,2-Dichloroethylene, ug/	/1 <1.0	
1,2-Dichloropropane, ug/l	<1.0	
Cis-1,3-Dichloropropene, ug/l	<1.0	
Trans-1,3-Dichloropropene, ug/1	<1.0	
Methylene Chloride, ug/l	<1.0	
1,1,2,2-Tetrachloroethane, ug/	<1.0	



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LOG NO: SE-00011

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Example Client 333 Main St. Savannah, GA 31404

> Project: SL I Report Sampled By: Client

REPORT OF RESULTS		Page 2
LOG NO SAMPLE DESCRIPTION , LIQUID SAMPLES		
00011-1 Water Sample 1 (Collected 2-20-91)		
PARAMETER	00011-1	
Tetrachloroethene, ug/l	<1.0	
1,1,1-Trichloroethane, ug/l	<1.0	
1,1,2-Trichloroethane, ug/l	<1.0	
Trichloroethene, ug/l	<1.0	
Trichlorofluoromethane, ug/l	<1.0	
Vinyl Chloride, ug/l	<1.0	
Purgeable Aromatics (602/8020)		
Benzene, ug/l	<1.0	
Chlorobenzene, ug/l	<1.0	·
1,2-Dichlorobenzene, ug/l	<1.0	
1,3-Dichlorobenzene, ug/l	<1.0	
1,4-Dichlorobenzene, ug/l	<1.0	
Ethylbenzene, ug/l	<1.0	
Toluene, ug/l	<1.0	
Xylenes, ug/l	<1.0	
Lead , ug/l	<5.0	

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LOG NO: SE-00011

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

Project: SL I Report

Sampled By: Client

REPORT OF RESULTS

LOG NO	SAMPLE DESCRIPTION , QC REPORT	FOR LIQUID SAMPLES		
				
	Method Blank			
	Laboratory Control Standard (LC	S) % Recovery		
	Precision (% RPD from LCS)			
PARAMETER			00011-3	00011-4
Durgeable W	(alocarbons (601/8010)			
-	oromethane, ug/l	<1.0		
Bromoform,	. 2.	<1.0		
Bromometha	•	<1.0		2
	rachloride, ug/l	<1.0		
Chlorobenz		<1.0	97 %	
Chloroetha		<1.0	J, •	4., 6
	hylvinyl Ether, ug/l	<1.0		
Chloroform	· ·	<1.0		
Chlorometh		<1.0		
	oromethane, ug/l	<1.0		
	probenzene, ug/l	<1.0		
•	probenzene, ug/l	<1.0		
	probenzene, ug/l	<1.0		
	fluoromethane, ug/l	<1.0		
1,1-Dichlo	proethane, ug/l	<1.0		
1,2-Dichlo	proethane, ug/l	<1.0		
1,1-Dichlo	proethene, ug/l	<1.0	99 🕏	2.9 %
Trans-1,2-	Dichloroethylene, ug/l	<1.0		
1,2-Dichlo	propropane, ug/l	<1.0		
Cis-1,3-Di	chloropropene, ug/l	<1.0	- 	
Trans-1,3-	Dichloropropene, ug/l	<1.0		



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LOG NO: SE-00011

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

> Project: SL I Report Sampled By: Client

REPORT OF RESULTS

Page 4

LOG NO	SAMPLE DESCRIPTION , QC REPORT	FOR LIQUID SAMPLES		
00011-3 00011-4	Method Blank Laboratory Control Standard (L Precision (% RPD from LCS)	CS) % Recovery		
PARAMETER		00011-2	00011-3	00011-4
Methylene	Chloride, ug/l	<1.0		
1,1,2,2-Te	etrachloroethane, ug/l	<1.0		
Tetrachloroethene, ug/l <1.0				
	chloroethane, ug/l	<1.0		
1,1,2-Tri	chloroethane, ug/l	<1.0	•	
Trichloro	ethene, ug/l	<1.0	101 %	1.3 %
Trichlorofluoromethane, ug/l <1.0		<1.0		
_	oride, ug/l	<1.0		
Purgeable 1	Aromatics (602/8020)			
Benzene, 1	_ ,	<1.0	99 🕏	1.4 %
Chloroben:	zene, ug/l	<1.0		
1,2-Dichlo	probenzene, ug/l	<1.0		
1,3-Dichlo	orobenzene, ug/l	<1.0		
1,4-Dichlo	orobenzene, ug/l	<1.0		
Ethylbenze	ene, ug/l	<1.0		
Toluene,	-	<1.0	103 %	2.7 %
Xylenes, w	ug/l	<1.0		•
Lead , ug/	1	<5.0	100 %	10 %

Methods: EPA 40 CFR Part 136

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FIGURE 12.4

EXAMPLE OF SL LEVEL II REPORT

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LOG NO: SE-00012

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

> Project: SL II Report Sampled By: Client

REPORT OF RESULTS

LOG NO	SAMPLE DESCRIPTION , LIQUID SAMPLES	DATE SAMPLED	
00012-1	Water Sample 1 (Collected 2-20-91)	02-20-91	
PARAMETER		00012-1	
	Halocarbons (601)		
Bromodich	loromethane, ug/l	<1.0	
Bromoform	, ug/l	<1.0	
Bromometha	ane, ug/l	<1.0	
Carbon Te	trachloride, ug/l	<1.0	
Chloroben	zene, ug/l	<1.0	
Chloroetha	ane, ug/l	<1.0	
2-Chloroe	thylvinyl Ether, ug/l	<1.0	
Chlorofor	m, ug/l	<1.0	
	hane, ug/l	<1.0	
	loromethane, ug/l	<1.0	
1,2-Dichle	orobenzene, ug/l	<1.0	
-	orobenzene, ug/l	<1.0	
	orobenzene, ug/l	<1.0	
	ifluoromethane, ug/l	<1.0	
=	oroethane, ug/l	<1.0	
	oroethane, ug/l	<1.0	
	oroethene, ug/l	<1.0	
	-Dichloroethylene, ug/l	<1.0	
-	oropropane, ug/l	<1.0	
	ichloropropene, ug/l	<1.0	
	-Dichloropropene, ug/l	<1.0	
-	Chloride, ug/l	<1.0	
1,1,2,2-T	etrachloroethane, ug/l	<1.0	

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LOG NO: SE-00012

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

> Project: SL II Report Sampled By: Client

REPORT OF RESULTS

LOG NO	SAMPLE DESCRIPTION , LIQUID SAMPLES		DATE SAMPLED
	Water Sample 1 (Collected 2-20-91)		02-20-91
PARAMETER		00012-1	
	oethene, ug/l	<1.0	
1,1,1-Tric	hloroethane, ug/l	<1.0	
1,1,2-Tric	hloroethane, ug/l	<1.0	
Trichloroe	thene, ug/l	<1.0	
Trichlorof	luoromethane, ug/l	<1.0	
Vinyl Chlo	ride, ug/l	<1.0	
Surrogate	- Bromochloromethane, ug/l	94 🕏	
Date Analy	zeđ	02.22.91	
Purgeable A	romatics (602/8020)		
Benzene, u	g/l	<1.0	
Toluene, u	g/l	<1.0	
Ethylbenze	ne, ug/l	<1.0	
Total Xyle	nes, ug/l	<1.0	
Methyl-Ter	t-Butyl-Ether (MTBE), ug/l	<1.0	
Total Vola	tile Organic Aromatics, ug/l	<1.0	
Surrogate	- a,a,a-Trifluorotoluene , ug/l	97 🕏	
Date Analy		02.22.91	
Lead			
Lead , ug/	1	<5.0	
Date Analy		02.22.91	



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LOG NO: SE-00012

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Example Client 333 Main St. Savannah, GA 31404

> Project: SL II Report Sampled By: Client

REPORT OF RESULTS

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CAVAMMAN I ADOCATORICE

LOG NO S	AMPLE DESCRIPTION , QC REPORT FO	R LIQUID SAMPLES		
00012-4 P:	ethod Blank aboratory Control Standard (LCS) recision (% RPD from LCS)	% Recovery		
PARAMETER			00012-3	
_	ocarbons (601) omethane, ug/l	<1.0	•	
Bromoform, u		<1.0		
Bromomethane	_	<1.0		
	chloride, ug/l	<1.0		• • •
Chlorobenzen		<1.0	108 %	2.2 %
Chloroethane	, ug/l	<1.0		
2-Chloroethy	lvinyl Ether, ug/l	<1.0		•
Chloroform,	ug/l	<1.0	••-	
Chloromethan	e, ug/l	<1.0		
Dibromochlor	omethane, ug/l	<1.0		
•	benzene, ug/l	<1.0	• • •	•
•	benzene, ug/l	<1.0		
•	benzene, ug/l	<1.0		
	uoromethane, ug/l	<1.0		
1,1-Dichloro	_	<1.0		
1,2-Dichloro		<1.0		
1,1-Dichloro			100 %	
	chloroethylene, ug/l	<1.0		
	propane, ug/1	<1.0		•
·	loropropene, ug/l	<1.0		
rrans-1,3-D1	chloropropene, ug/l	<1.0		

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Example Client 333 Main St. Savannah, GA 31404

Project: SL II Report

Sampled By: Client

REPORT OF RESULTS

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LOG NO	OG NO SAMPLE DESCRIPTION , QC REPORT FOR LIQUID SAMPLES			
00012-2 Method Blank 00012-3 Laboratory Control Standard (LCS) % Recovery 00012-4 Precision (% RPD from LCS)				
PARAMETER		00012-2	00012-3	00012-4
Methylene	Chloride, ug/l	<1.0		
1,1,2,2-Te	trachloroethane, ug/l	<1.0		
Tetrachlor	oethene, ug/l	<1.0		• • •
1,1,1-Tric	hloroethane, ug/l	<1.0		
1,1,2-Tric	hloroethane, ug/l	<1.0		
Trichloroe	thene, ug/l	<1.0	93 🕏	1.3 %
Trichlorof	luoromethane, ug/l	<1.0	•	
Vinyl Chlo	ride, ug/l	<1.0		
Surrogate - Bromochloromethane, ug/l		98 %	97 🕏	4.5 %
Date Analyzed		02.22.91		
Purgeable A	romatics (602/8020)			
Benzene, u	g/l	<1.0	99 🕏	4.5 %
Toluene, u	g/l	<1.0	102 %	6.7 🕏
Ethylbenze	ne, ug/l	<1.0		
Total Xyle	nes, ug/l	<1.0		
Methyl-Ter	t-Butyl-Ether (MTBE), ug/l	<1.0		
Total Vola	tile Organic Aromatics, ug/l	<1.0		
Surrogate	- a,a,a-Trifluorotoluene , ug/l	99 🕏	104 %	5.9 🕏
Date Analy	zed	02.22.91		
Lead				
Lead , ug/	1	<5.0	90 🕏	10 %
Date Analy	zed	02.22.91		•••

Methods: EPA 40 CFR Part 136

Case Narrative - No QC problems were encountered.



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FIGURE 12.5

EXAMPLE OF SL LEVEL III REPORT

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LOG NO: SE-00015

Received: 21 FEB 91

Example Client 333 Main St. Savannah, GA 31404

Project: SL III Report (CLP Type)

Sampled By: Client

REPORT OF RESULTS

00015-1 Water Sample 1 (Collected 2-20-	91)
PARAMETER	00015-1
Purgeable Halocarbons (601)	
Bromodichloromethane, ug/l	1.00
Bromoform, ug/l	1.00
Bromomethane, ug/l	1.00
Carbon Tetrachloride, ug/l	1.00
Chlorobenzene, ug/l	1.00
Chloroethane, ug/l	1.00
2-Chloroethylvinyl Ether, ug/l	1.00
Chloroform, ug/l	1.00
Chloromethane, ug/l	1.00
Dibromochloromethane, ug/l	1.00
1,2-Dichlorobenzene, ug/l	1.00
1,3-Dichlorobenzene, ug/l	1.00
1,4-Dichlorobenzene, ug/l	1.00
Dichlorodifluoromethane, ug/l	1.00
1,1-Dichloroethane, ug/l	1.00
1,2-Dichloroethane, ug/l	1.00
1,1-Dichloroethene, ug/l	1.00
Trans-1,2-Dichloroethylene, ug/l	1.00
1,2-Dichloropropane, ug/l	1.00
Cis-1,3-Dichloropropene, ug/l	1.00
Trans-1,3-Dichloropropene, ug/l	1.00
Methylene Chloride, ug/l	1.00
1,1,2,2-Tetrachloroethane, ug/l	1.00

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LOG NO: SE-00015

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Example Client 333 Main St. Savannah, GA 31404

Project: SL III Report (CLP Type)

Sampled By: Client

REPORT OF RESULTS

LOG NO SAMPLE DESCRIPTION , LIQUID SAMPLES		
and no branch basedarines, bigots official		
00015-1 Water Sample 1 (Collected 2-20-91)		
PARAMETER	00015-1	
		· · · · · · · · · · · · · · · · ·
Tetrachloroethene, ug/l	1.00	
1,1,1-Trichloroethane, ug/l	1.00	
1,1,2-Trichloroethane, ug/l	1.00	
Trichloroethene, ug/l	1.00	
Trichlorofluoromethane, ug/l	1.00	
Vinyl Chloride, ug/l	1.00	
Surrogate - Bromochloromethane, ug/l	94 %	
Date Analyzed	02.22.91	
Purgeable Aromatics (602/8020)		
Benzene, ug/l	1.00	
Toluene, ug/l	1.00	
Ethylbenzene, ug/l	1.00	
Total Xylenes, ug/l	1.00	
Methyl-Tert-Butyl-Ether (MTBE), ug/l	1.00	
Total Volatile Organic Aromatics, ug/l	1.00	
Surrogate - a,a,a-Trifluorotoluene , ug/l	97 %	
Date Analyzed	02.22.91	
Lead		
Lead , ug/l	5.0U	
Date Analyzed	02.22.91	

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Example Client 333 Main St. Savannah, GA 31404

Project: SL III Report (CLP Type)

Sampled By: Client

REPORT OF RESULTS

LOG NO SA	AMPLE DESCRIPTION , QC REPOR	RT FOR LIQUID SAMPLES		
00015-3 La	ethod Blank aboratory Control Standard recision (% RPD from LCS)	(LCS) % Recovery		
PARAMETER			00015-3	00015-4
Purgeable Hale				
-	omethane, ug/l	1.00		
Bromoform, u		1.00		
Bromomethane		1.00	• • •	
	chloride, ug/l	1.00		
Chlorobenzen	_	1.00	108 %	2.2 🕏
Chloroethane		1.00		-+-
2-Chloroethy	lvinyl Ether, ug/l	1.00		
Chloroform,	ug/l	1.00		
Chloromethan	e, ug/l	1.00		
Dibromochlor	omethane, ug/l	1.00		
1,2-Dichloro	benzene, ug/l	1.00		
1,3-Dichloro	benzene, ug/l	1.00		
1,4-Dichloro	benzene, ug/l	1.00		
Dichlorodifl	uoromethane, ug/l	1.00		
1,1-Dichloro	ethane, ug/l	1.00		• • • •
1,2-Dichloro	ethane, ug/l	1.00		
1,1-Dichloro	ethene, ug/l	1.00	100 %	
Trans-1,2-Di	chloroethylene, ug/l	1.00		
1,2-Dichloro	propane, ug/l	1.00		
	loropropene, ug/l	1.00		
Trans-1,3-Di	chloropropene, ug/l	1.00		

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LOG NO: SE-00015

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Example Client 333 Main St. Savannah, GA 31404

Project: SL III Report (CLP Type)

Sampled By: Client

REPORT OF RESULTS

LOG NO	SAMPLE DESCRIPTION , QC REPORT FOR I	LIQUID SAMPLES		
00015-3	Method Blank Laboratory Control Standard (LCS) % Precision (% RPD from LCS)	Recovery		
PARAMETER		00015-2	00015-3	00015-4
Methylene	Chloride, ug/l	1.00		
1,1,2,2-Te	etrachloroethane, ug/l	1.0U		
	roethene, ug/l	1.00		
1,1,1-Tric	chloroethane, ug/l	1.00		
1,1,2-Tric	chloroethane, ug/l	1.00		
Trichloro	ethene, ug/l	1.00	93 %	1.3 %
Trichloro	fluoromethane, ug/l	1.00		
Vinyl Chlo	oride, ug/l	1.00	•	
Surrogate	- Bromochloromethane, ug/l	98 %	97 %	4.5 %
Date Analy	yzed	02.22.91		
Purgeable i	Aromatics (602/8020)			
Benzene,	ug/l	1.00	99 🕻	4.5 %
Toluene,	ug/l	1.00	102 %	6.7 🕏
Ethylbenze	ene, ug/l	1.0U		
Total Xyl	enes, ug/l	1.00	•	
Methyl-Te:	rt-Butyl-Ether (MTBE), ug/l	1.00		
Total Vol	atile Organic Aromatics, ug/l	1.00		
Surrogate	 a,a,a-Trifluorotoluene , ug/l 	99 🖁	104 %	5.9 🕏
Date Analy	yzed	02.22.91	• • •	
Lead				
Lead , ug		5.0 0	90 %	10 %
Date Anal	yzed	02.22.91		

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Example Client 333 Main St. Savannah, GA 31404

Project: SL III Report (CLP Type)

Sampled By: Client

REPORT OF RESULTS

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LOG NO	SAMPLE DESCRIPTION , QC REPORT FOR LIQU			
00015-5 00015-6	Matrix Spike (% Rec) Matrix Spike Duplicate (% Rec)			
PARAMETER		00015-5	00015-6	
				•
Purgeable H	alocarbons (601)			
Chlorobenz	ene, mg/l	98 🕏	100 %	
1,1-Dichlo	roethene, mg/l	104 %	102 %	
Trichloroe	thene, mg/l	95 %	98 %	
Surrogate	- Bromochloromethane, mg/l	99 🕏	98 %	
Purgeable A	romatics (602/8020)			
Benzene, u	g/1	94 %	96 %	
Toluene, u	- '.	99 🕏	101 %	
	- a,a,a-Trifluorotoluene , ug/l	102 %	103 %	
Lead	_, _, _ , ,,,,,			
Lead , ug/	1	98 🕏	100 %	

Methods: EPA 40 CFR Part 136 See attached data package.



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13.0 CORRECTIVE ACTION

Corrective action will be initiated when data are determined to be questionable or QC criteria are out of control. For routine operational problems, the analysts correct the problem and note the problem/corrective action on the run log or bench data sheet. A Corrective Action Report (CAR) is not necessary unless the problem is recurring.

When formal corrective action is required, a Corrective Action Report (CAR) is prepared on the CAR form (Figure 13.1). CAR are required for:

- 1. Chronic problems which could affect data quality or production and are due to equipment or facility disrepair or inadequacy, improper training, employee attitude or ineptness, supply, reagent or standard quality, SOP inadequacy or error, or any other problems which could be corrected by management. CARs for this type of problem should be prepared by the analyst and channelled through the department manager/lab manager to the laboratory director. The box by "Request Lab Director's Attention" should be checked and final action should be taken by the lab director.
- 2. For uncorrectable nonconformance problems which are noted by an asterisk (*) in Table 13.1 which could affect the quality of report data, corrective action is initiated by the analyst or department manager. Before a CAR is prepared, the analyst/department manager will review raw data calculations, procedures, methods, operating conditions of the instrument and all data available. If this does not resolve the problem, analysis of the batch (samples plus QC samples) is repeated provided sufficient sample is available. If data are submitted in cases where QC is not in compliance, this is documented in a case narrative which is part of the data reports. The action must be approved by the project manager who submits the report.
- When QA problems are discovered during data review, system audits, performance audits, audit sample results, client inquiries, external data review or validation, or audits, a CAR is prepared by the QA manager, and is filed for use in QA reports to management.
- 4. When QA data exceed the criteria in Table 13.2, the analyst or department manager initiates a CAR.

All CARs are filed in the departmental corrective action notebook which has a pending and completed section. Follow-up is checked weekly by the department manager and monthly by the QA manager.

If warning limits are exceeded, the department manager/supervisor points this out to the respective supervisors or chemists who attempt to define and correct the problem.

Savannah Laboratories will abide by any corrective action deemed necessary by all pertinent agencies.

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FIGURE 13.1

CORRECTIVE ACTION REPORT (CAR)

Date Prepared:	Sample ID:)	SL P	roject ID:
Analysis:		Date of Analysis	:
Analyst: Depa	rtment Manager:	Proj	ect Manager:
Description of Nonconformance/Condi	tion:		
Corrective Action Implemented:			
On Manage and Tarked and		Date of America	
QA Manager's Initials:			
□ Request Lab Director's attention			
□ Request Project Manager's attent			
Date Corrective Action Implemented		By:	
Corrective Action Follow-up/Commen	ts:		
	-		
Corrective Action Completed: Depa	rtment Manager's i	nitials:	Date:
QA Manager's Initials:		Date:	

Copies of this report should be filed in the laboratory Corrective Action Notebook.

TABLE 13.1

CORRECTIVE ACTION

	QC Activity	Acceptance Criteria	Recommended Corrective Action
*	GC/MS tuning or ICP/AA	Per SOPs or Chapter 9.0	Do not analyze samples unless criteria are met.
•	Initial calibration standards	Per SOPs or Chapter 9.0	Reanalyze standards. If still unacceptable, remake standards or instrument corrections.
*	QC check/continuing calibration standard	Per SOPs, See Chapter 9.0	Reanalyze standard. If still unacceptable, remake standards, or recalibrate.
*	Method blank	< PQL (for CLP procedures, use SOW guidelines)	Reanalyze blank. If problem, determine source of contamination. If necessary or possible, redigest/extract batch and reanalyze.
*	Surrogate recovery (GC/MS semivolatiles)	Tables 5.1 and 5.2. One acid and one base may be out of criteria.	Follow SW-846 method or CLP guidelines.
*	Surrogate recovery (GC/MS volatiles)	O outside criteria in Tables 5.1 and 5.2	Follow SW-846 method or CLP guidelines.
	Surrogate recovery GC or LC	Tables 5.1 and 5.2	Criteria advisory only; check for possible matrix interferences or other causes.
	Matrix spike recoveries	Tables 5.1 and 5.2	Criteria advisory only; check for possible matrix interferences or other causes.
•	Lab control standard recoveries	Tables 5.1 and 5.2	Check calculations, reanalyze standards, and if necessary or possible, redigest or extract batch and reanalyze.

^{*} If criteria cannot be met, a corrective action report (CAR) must be prepared and approved by QA manager and project manager.

TA	BI	Æ	13	- 2

Corrective Action Report Criteria for Control Charts

Criteria	Corrective Action
A point outside ± 3 standard deviations	Check calculations. Report the deviation and results of preliminary investigation to the division manager, and the QA manager, who will decide jointly what action to take. Complete the Corrective Action Report and submit it to the department manager and QA manager for approval.
Obvious shift in the mean	Check calculations, data entry, standards, instrument, calibrations, etc. Document results in a Corrective Action Report.
Any 8 consecutive points are on the same side of the mean	Check accuracy of data entry and calculations. Document results in a Corrective Action Report. Have the report approved by the department, QA, and project managers.
Any 6 consecutive points are such that each point is larger (smaller) than its immediate predecessor	Check accuracy of data entry and calculations. Document results in a Corrective Action Report. Have the report approved by the department, QA, and project managers.
Any obvious cyclic pattern is seen in the points	Check accuracy of data entry and calculations. Document results in a Corrective Action Report. Have the report approved by the department, QA, and project managers.

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14.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits are performed in each laboratory throughout the year.

14.1 Internal System Audits

14.1.1 Annual Corporate Audits

On an annual basis, an on-site systems audit is conducted on all aspects of the laboratory and field operations at each facility. This audit is coordinated by the president and is conducted by a multiperson audit team made up of individuals with expertise in the organic, inorganic, QA, project custody, data management, and field sampling areas, the corporate safety director, and a representative from the business office. This onsite audit may be supplemented by review of reports and QA data in the LIMS network and review of selected data packages. An audit report is issued by the team, to the president within two weeks of completion of the audit and a copy is provided by the QA manager to the lab director.

The annual system audits consist of an examination of laboratory procedures and documentation to ensure that the entire laboratory is being operated according to established protocol. The auditors will ensure that the proper frequency of quality control standards, spikes, duplicates, etc., are incorporated with each sample analytical run, and all results are documented, up to date, and accessible for review. Control charts are checked to ensure their proper maintenance. Calculations are spot checked and data procedures are reviewed to ensure SOPs are being followed, and special attention is given to calibration procedures. The systems audit check also ascertains whether proper documentation exists to trace working analytical standards back to stock standards. Finally, analysts' techniques are evaluated against techniques as defined in the SOPs, the SL Training SOP, and recognized good laboratory practices.

The QA manager and lab director respond to the audit and are responsible for following up on required corrective action.

14.1.2 Quarterly Internal Audits

Quarterly audits are conducted by lab QA managers. Results of these audits are used in preparation of quarterly reports to management. Responses and follow-up corrective actions are addressed by department managers and monitored by the lab QA manager.

14.1.3 Internal Systems Audit Checklist

Figure 14.1 is a page from a laboratory checklist used to conduct an internal systems audit at Savannah Laboratories. This particular example contains quality assurance questions directed to the custody section of the laboratory.

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14.2 External System Audits

Each laboratory is certified by several state agencies and governmental or private certification programs. The laboratories submit to external onsite audits by these certifying agencies or organizations.

Field system audits are performed periodically by various federal and state regulatory agencies. Field sampling and documentation procedures are examined to insure sampling is performed according to the protocols established in this document.

14.3 Performance Audits

14.3.1 Internal Performance Audits

The laboratory QA manager periodically schedules blind audit samples into the work flow. Major methods are tested by at least two internal audit samples annually. Audit samples are treated as actual samples and are logged into the LIMS. Results are entered into the LIMS and summarized by the QA manager and presented to the department managers, lab manager, and lab director. Problem results are addressed in corrective action and/or quarterly QA reports to management.

14.3.2 External Performance Audit

All facilities participate in the following performance evaluation audits quarterly:

- U. S. EPA Water Supply Study (WS Series).
- U. S. EPA Water Pollution Study (WP Series).

Additionally, the laboratories participate in several regulatory agency, certifying group, or client requested performance audits. Results from these performance audits are included in quarterly QA reports to management.

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specific information for either the performance or systems audits, significant QA/QC problems, and corrective action status as described in Appendix D. If no project audits are performed and no significant QA/QC problems occur for the duration of a project requiring a DER QA report, a letter stating these facts will be submitted in lieu of the QA report.

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16.0 PERSONNEL QUALIFICATIONS

Listing of Technical Employees:

SAVANNAH DIVISION

EMPLOYEE	DEGREE	TITLE
James W. Andrews	Ph.D.	President
Janette D. Long	BS	Vice President/Lab Director
Jay W. Andrews	BS	Controller/Secretary/Treasurer
Alan C. Bailey	Ph.D.	Quality Assurance Manager
Steven J. White	BS	Project Manager
Linda A. Wolfe	BS	Project Manager
Beverly Hughes	BS	Project Manager
Larry E. Phillips	BS	Computer Manager
Wayne Robbins	BS	Air Analysis Manager
Penny Carter	BS	Computer Programmer
Sheila B. Hoffman	BS	Data Manager
Virginia Baisden	MS	Field Sampling Manager
Derrick M. Simons	BS	Laboratory Manager
Ernest B. Walton	BS	Corporate Inorganic Manager
Karla J. Bier	BS	SG Manager
Karri L. Derr	BS	VG Manager
Mike Salum	BS	Organic Extraction Manager
Myron J. Young	BS	VM Manager
Paul E. Meyers	BS	Chemist/Corporate Safety Director
Kenneth R. Aegan	BS	Chemist
Lisa D. Aegan		Technician
Robert Bearden		Analyst
Jesse B. Blackwell, Jr.	BS	Analyst
Bernetha Brayboy	BS	Analyst
Nancy Brown	BS	Analyst
Laura R. Bulluck	BS	Analyst
Hsaio Lan Chang	MS	Analyst
Glen A. Coder	BA	Analyst
Katherine M. Cook	BS	Chemist
Nannette H. Dasher	BS	SM Supervisor
Kelly T. Durden	BS	Analyst
Dagmar Goley	BS	Inorganic CLP Coordinator
Sandra Grovenstein	BS	SM Supervisor
Robert K. Hamilton	BS	Chemist
Phillip S. Harvey	BS	Chemist

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SAVANNAH DIVISION (Continued)

EMPLOYEE	DEGREE	TITLE
Reginald H. Hendrix		Technician
Theresa Hornsby	BS	Analyst
Daphne Hughes	BS	Analyst
James Johnson		Analyst
Bernard Kirkland	BS	Chemist
G. Anthony Lowman	BS	Analyst
Carl E. Manning	BS	Chemist
Deborah McDonald		Analyst
Sarah A. McMillan	BA	Analyst
Kimberley D. McNeill	BS	Analyst
Michael W. Mullenix		Analyst
Susan K. Norwood	BS	Metals ICP/AA Supervisor
Everett W. Owens	BS	Chemist
J. Robert Paddison, Jr.	MS	VM Supervisor
Ruth D. Rankin	BS	Digestion Supervisor
Lorene E. Reeves	BS	Chemist
Michelle L. Long	BS	Chemist
Cynthia E. Schlag	BS	Chemist
Charles W. Schuman	BS	Analyst
Elizabeth R. Sicay	BA	Analyst
Julie L. Silvey	BS	Chemist
Angela F. Stewart	BS	Chemist
Melanie T. Walsh	BS	Chemist
Ashton A. Waters	BS	Analyst
Angela M. Weimerskirk	BS	General Lab Supervisor
Barry L. Williams	BA	Analyst
Millicent A. Williams	BS	Analyst
Laura Willman	BS	Chemist
Jeff Wilmoth	BS	Analyst
Angela Zealy	BS	Chemist

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TALLAHASSEE DIVISION

EMPLOYEE	DEGREE	TITLE
Thomas L. Stephens	BS	Vice President/Project Manager
Janet B. Pruitt	MS	Lab Director/Project Manager
Wayne Word	BS	Project Manager
C. Henry Beauchamp	BS	Laboratory Manager
Elizabeth L. Schneider	BS	Quality Assurance Manager
Todd A. Baumgartner	BS	Inorganic Section Manager
David A. Karns	BS	GC/MS Manager
Bernard Ash	BS	Chemist
Brian Corbin	BS	Chemist
Robert D. Driver	BS	Analyst
Sus an Harrison	BS	Chemist
D. Wayne Higgins	BS	Analyst
Mavis LaBounty	BS	Analyst
Andre Miley	BS	Analyst
Richard Orr	BA	Chemist
Tim Preston	BS	Chemist
Paul Rygiel	BS	Chemist
Deborah Sherwin	BS	Biological/General Supervisor
Richard Stephens	BS	Volatiles Section Manager
Martin Thomas		Technician
Dana B. Till	BS	Chemist

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MOBILE DIVISION

EMPLOYEE	DEGREE	TITLE
Jesse L. Smith	BS	Lab Director/Project Manager
James M. Nance, Jr.	MS	Project Manager
Michele H. Lersch	BS	Quality Assurance Manager
Van Pham	Ph.D.	Laboratory Manager
Cora Mae Pate	BS	Volatiles Supervisor
Bruce H. Barrett	BS	Chemist
Rebecca Bowen	Ph.D.	Analyst/Chemical Hygiene Officer
Panda W. Carter	BS	Metals Analyst
Chris Cook	BA	Field Manager
Cedric Crawley		Technician/Extraction Group Leader
Sheryl S. Fuller	BS	Chemist
Stephanie Jones	BS	Field Scientist
Shao-Wei Li	Ph.D.	Chemist
Katherine W. Morgan	BS	Sample Coordinator
Edward Oetken	BS	Analyst
Tracy Owens	BS	Metals Supervisor
Michael Reardon		Technician
Sonya Reynolds	BS	Analyst
Letitia Saunders	BA	Analyst
Nan Scarbrough	BS	Analyst
John Sh oemaker		Technician
John Sims	BS	Analyst
David Sweet		Technician
Rhoda L. Smith		Data Coordinator
Virginia Vasquez	BS	Chemist
Cynthia Wilson	BS	Chemist
Joyce Zatarain		Technician

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DEERFIELD BEACH DIVISION

EMPLOYEE	DEGREE	TITLE
Paul K. Canevaro	BS	Lab Director/Project Manager
Marianne J. Walker		Sample/Data Manager
Rhonda Moll	BS	Project Managaer
Kathy C. Irminger	BA	Quality Assurance Manager
Phill Taylor, Jr.		Field Coordinator
Kimberly L. Ambrisco-Kostze	er BS	SG Manager
Linda Backus	BS	Chemist
David Graham	BA	Analyst
Therona James	BA	Chemist
Catherine Katsikis	BS	Chemist
Nanette Kendall	BA	Chemist
Kim Puhl	BS	Chemist
Eric Schinsing	AA	Technician
Alicia Stewart	AA	Technician
Lawrence Teich	BS	Chemist
Mary Valest	BS	Chemist
Carol-Ann Vassell	BS	Chemist
Janice Wiltshire		Data Coordinator

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TAMPA BAY DIVISION

EMPLOYEE	DEGREE	TITLE
Kathy Sheffield	BS	Lab Director/Project Manager
Andre Rachmaninoff	BA	Project Manager
Dominic Fralli	MS	Project Manager
Inas M. Sobky	BS	Quality Assurance Manager
Tracy Botto	BS	Inorganics Section Manager
Linda Dowd	BS	Analyst
Chris E. Harris		Field Coordinator
Carl John Hoover, Jr.	BS	Chemist
Cheryl L. Howard	BA	Chemist
Antonius Lebrun	BS	Chemist
Marsha Martinovich	BA	Analyst
Natalie Park	BS	Analyst
Talicia C. Smith	BS	Chemist
Tayseer Zayan	BS	Chemist

Resumes of professional personnel are included in the following pages.

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JAMES W. ANDREWS

President - Project Manager, Savannah Division

Dr. Andrews holds a B.S. degree in chemistry and an M.S. and Ph.D. in nutritional physiology from the University of Georgia. Dr. Andrews' specialty is aquatic chemistry and biochemical and physiological effects of chemicals on animals.

In 1962, Dr. Andrews began his professional career as an environmental chemist with the research division of Continental Forest Industries. During this employment, his duties involved developing techniques for reducing water and air pollution from pulp and paper mills and water quality evaluations of streams.

From 1963 to 1968, he was a research assistant and lecturer at the University of Georgia. As part of this work, he was assigned to special projects at the Hormel Institute in Austin, Minnesota, and at INCAP in Guatemala City, Guatemala.

In 1968, he became one of the initial scientists at the Skidaway Institute of Oceanography in Savannah, Georgia. During his tenure at Skidaway Institute, he was the principal investigator of many biological, physiological and fish cultural studies. Dr. Andrews is the author of more than 70 research papers in the aquatic field. In 1976, he was selected to be a member of a National Academy of Sciences subcommittee on aquatic nutrition.

For several years, Dr. Andrews has worked as a volunteer with the Community Cardiovascular Council of Savannah and Dr. Curtis Hames of the Evans Cardiovascular Project. In this capacity, he has become involved in several multi-national research projects which were designed to relate environmental and dietary exposure to cardiovascular health. This work has lead to several scientific publications on the effect of environmental exposure to heavy metals on human health in the high cardiovascular disease area of the southeastern United States.

Dr. Andrews has been a private consultant on environmental and water quality aspects of the coastal southeast since 1969. Since 1975, he has been the President of Savannah Laboratories and Environmental Services, Inc. His primary functions at Savannah Laboratories are the evaluation and interpretation of data and responding to the advisory needs of engineers, environmental specialists, legal experts, and production personnel, as well as supervising bioassay/bioaccumulation and environmental studies.

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JANETTE D. LONG

Vice President - Project Manager/Laboratory Director, Savannah Division

Ms. Long has a B.S. degree in chemistry and 16 years experience in the analysis and data review of water, soil, biological and other environmental matrices. Prior to her association with Savannah Laboratories and Environmental Services, Inc., she was a research chemist with the University of Georgia Experiment Station evaluating biological tissues, enzymes and water samples. During her involvement with the Experiment Station, she co-authored several research papers in the aquatic field.

For several years, she assisted the Community Cardiovascular Council of Savannah as a volunteer research chemist. During this time, she assisted in the research effort as well as co-authored several publications concerning the epidemiological aspects of heavy metal exposure on human health in the southeastern United States. Ms. Long has been active in the American Chemical Society activities in the environmental area. She has held several offices in the organization, including President of the Coastal Empire Region.

Ms. Long began her association with Savannah Laboratories in 1975, and as a project manager, has worked closely with clients to review site-specific project plans, generic QA project plans, project regulatory concerns and to ensure that the analyses recommended will provide the desired data and QA/QC requirements requested by the client. She has been responsible for proposal preparation and project management for numerous RCRA, NPDES, environmental impact assessments and other related projects.

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STEVEN J. WHITE

Project Manager, Savannah Division

Mr. White has a B.S. degree in chemistry and seven years of experience with Savannah Laboratories and Environmental Services, Inc. As a project manager, he serves as a point of contact for clients needing technical support in the areas of sampling, analysis, and the evaluation of laboratory results. He has extensive experience in the analysis of environmental pollutants using gas chromatography, GC/MS, and atomic absorption techniques. He possesses comprehensive knowledge of EPA procedures for the determination of pesticides, herbicides, PCBs, PAHs, base/neutral and acid extractable organics in various sample matrices.

Prior to his association with Savannah Laboratories in 1984, he pursued graduate studies at the Institute of Paper Chemistry and the University of Georgia. He has participated in EPA-sponsored workshops for pesticide residue analysis and has attended seminars on numerous topics in environmental analysis. Mr. White is an active member of the American Chemical Society.

LINDA A. WOLFE

Project Manager, Savannah Division

Ms. Wolfe has a B.S. degree in chemistry and a B.S. degree in biology with four years laboratory experience in the determination of metals and one year in semivolatiles GC section in the analysis of samples for pesticides, PCBs, and herbicides.

Prior to her association with Savannah Laboratories and Environmental Services, Inc., Ms. Wolfe was production specialist and wastewater lab specialist at SCM Corporation for two years. She joined Savannah Laboratories in 1985 as a laboratory metals analyst, and has done extensive work with inductively coupled plasma (ICP) spectroscopy and atomic absorption furnace and flame spectroscopy. Ms. Wolfe is trained in clean room techniques and trace level extractions. She is experienced with all inorganic methods contained in EPA 600/4-79-020, SW-846, and CLP documents.

In her current role, Ms. Wolfe provides assistance for clients in areas of analysis and evaluation of laboratory results.

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BEVERLY HUGHES

Project Manager, Savannah Division

Ms. Hughes has a B.S. degree in biology/natural sciences and seven years experience in the analysis of environmental pollutants by gas chromatography. Prior to her association with Savannah Laboratories and Environmental Services, Inc. in 1987, she was an environmental specialist at the SCM Corporation.

Since joining Savannah Laboratories, Ms. Hughes has worked extensively with semivolatile gas chromatography overseeing pesticides, PCBs, herbicides, and other semivolatile organic compounds. Her work has given her extensive experience with SW-846, 40 CFR, and CLP protocols.

In her current role, Ms. Hughes provides assistance to clients in the areas of analysis and evaluation of laboratory results.

ALAN BAILEY

Laboratory QA Manager, Savannah Division

Dr. Bailey holds a B.S. degree in chemistry and biology from the University of Georgia and a Ph.D. in analytical chemistry from Clemson University. Between his undergraduate and graduate studies, he worked as a chemist at Union Carbide Agricultural Products Company.

Dr. Bailey's graduate research involved new approaches to the study of chemical exchange across the sediment water interface in both marine and freshwater systems. As a graduate student, he worked two summers in collaborative research in Environmental Research Division at Argonne National Laboratories. Also, while at Clemson, he taught laboratories in freshman chemistry, quantitative analysis, and instrumental analysis, devising and implementing several new experiments for the analytical teaching laboratories. Dr. Bailey is a member of the American Chemical Society and the International Association for Great Lakes Research.

Dr. Bailey began his association with Savannah Laboratories in 1989. As manager of the General Laboratory section of the Savannah Division, Dr. Bailey is responsible for personnel management, production, and quality control for a wide variety of analyses. The General Laboratory section includes nutrients, cyanide, microbiological parameters, BOD, COD, TOC, TOX, and many other physical and chemical parameters.

Currently, he is QA manager and, among other duties, is responsible for internal systems audits and performance evaluations, certifications, and updates/revisions to Savannah Laboratories' QA plan. Dr. Bailey is a member of the American Chemical Society and the International Association for Great Lakes Research.

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DERRICK M. SIMONS

Lab Manager, Savannah Division

Derrick M. Simons obtained a B.S. degree from the University of Florida in 1982, with majors in both chemistry and microbiology & cell science. While an undergraduate at the University of Florida, Mr. Simons worked as a research assistant in natural products chemistry.

From 1982 to 1986, Mr. Simons served as a chemist in a commercial environmental testing laboratory, where his duties included the determination of Pesticides, PCBs, Herbicides, Volatile and Semivolatile Organic Compounds by GC and GC/MS methodologies.

Mr. Simons was promoted in 1986 to GC and GC/MS group leader for both Volatile and Semivolatile Organic departments. In 1987, he was promoted to organics lab manager, supervising all GC, GC/MS, and organic extraction personnel where he obtained extensive knowledge of SW-846, 40 CFR, and CLP protocols.

Mr. Simons joined Savannah Laboratories in 1990 as Corporate Organic Manager and Savannah Division Organic Manager. His duties included responsibility for GC, GC/MS, and the organic extraction sections. As well, he was responsible for organic analytical method development, overseeing the training of new personnel, and supervising the maintenance and troubleshooting of GC and GC/MS instrumentation. He was promoted to Lab Manager in the summer of 1992. Additional responsibilities of this position include overall administrative responsibility for all technical lab personnel. As Corporate Organic Manager, he is responsible for preparing SOPs, establishing analytical and QA procedures, evaluating instrumentation, and coordinating production among the organic departments.

LARRY E. PHILLIPS

Corporate Computer Manager, Savannah Division

Mr. Phillips attended Armstrong State College where he received a B.S. degree in computer science. He has two years experience with Armstrong State College where he was responsible for maintaining system communications in addition to hardware maintenance and system backups.

Mr. Phillips joined Savannah Laboratories and Environmental Services, Inc. in 1988. He is responsible for maintaining all software and hardware associated with Savannah Laboratories' Laboratory Information Management System (LIMS). In addition, Mr. Phillips is responsible for new software development and testing along with maintaining data communications between Savannah Laboratories Corporate Headquarters and Tallahassee, Mobile, Tampa Bay, and Deerfield Beach divisions.

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WAYNE ROBBINS

Air Analysis Manager, Savannah Division

Mr. Robbins has a B.S. degree in chemistry and has been trained in analytical and quality control techniques for a wide variety of procedures. He is thoroughly familiar with EPA approved procedures and has attended several EPA training schools on analytical techniques. He is currently in charge of implementing protocols for the analysis of ambient air samples and is responsible for coordinating production, preparing SOPs, and evaluating methodology for this section.

Mr. Robbins has ten years experience in the analysis of environmental samples by EPA procedures.

ERNEST WALTON

Corporate Inorganic Manager/Inorganic Manager, Savannah Division

Mr. Walton has a B.S. degree in chemistry from Mercer University and began his association with Savannah Laboratories in 1983. His major area of concentration is the analysis of metals in ground water, biological tissues, sediments and estuarine water. He has been trained in clean room sample preparation techniques and has participated in various training courses of metal analysis utilizing inductively coupled plasma spectroscopy and atomic absorption methodology. He also has experience with various automated, semiautomated, and manual nutrient analysis systems.

Mr. Walton has been trained in quality control procedures for evaluating laboratory data and has attended the Waste Testing and Quality Assurance symposium in Washington, DC. He was responsible for the initial implementation of CLP protocol for the laboratory's inorganic section. Mr. Walton also pioneered the use of software by the laboratory for the production of CLP deliverables. As well as being thoroughly experienced with CLP protocol, Mr. Walton is knowledgeable in all the inorganic methods contained in EPA 600/4-79-020 and SW-846 documents. As Corporate Inorganic Manager, he is responsible for preparing SOPs, establishing analytical and QA procedures, evaluating instrumentation, and coordinating production among the inorganic departments.

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SHEILA B. HOFFMAN

Data Manager, Savannah Division

Ms. Hoffman has a B.S. degree from Georgia Southern University and began her career with Savannah Laboratories and Environmental Services, Inc. in 1982 as a sample coordinator.

She has been responsible for the development of the data management coordination of the laboratory as well as sample custodial responsibilities. Ms. Hoffman coordinates all project orders from the sample login to the computer project login. She interfaces with the clients and project managers to facilitate the data flow through the laboratory and coordinates client sample container requests. Ms. Hoffman has extensive experience with chain of custody for CLP projects and other client specific QA requirements.

VIRGINIA BAISDEN

Field Sampling Manager, Savannah Division

Ms. Baisden has B.S. and M.S. degrees in biology and more than 12 years experience in field sampling and biological and chemical analyses of samples.

Prior to her association with Savannah Laboratories and Environmental Services, Inc. in 1986, Ms. Baisden was employed by the Georgia Department of Natural Resources, Coastal Resources Division, where she was project leader of the Commercial Fisheries Program. While associated with the Coastal Resources Division, she authored several reports and publications of fisheries assessment studies. Ms. Baisden has worked with the Game and Fish Division where she identified zooplankton. She was a research assistant for the Environmental Protection Division on an estuarine water quality monitoring project.

Ms. Baisden's primary duties at Savannah Laboratories include responsibility for all biological and microbiological analyses and coordinating and supervising field sampling.

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MYRON J. YOUNG

Volatiles GC/MS Manager, Savannah Division

Mr. Young has a B.S. degree in chemistry and an A.S. degree in electronic engineering. Prior to his employment at Savannah Laboratories and Environmental Services, Inc. in 1987, he was employed as a chemist with Southeast Laboratories of Atlanta, Georgia.

Mr. Young manages the GC/MS volatiles section at Savannah Laboratories and is responsible for coordinating all QA/QC requirements for that department. He is thoroughly familiar with techniques for performing analyses on many different compounds and the operation of the GC and GC/MS instrumentation used to perform such analyses. He is familiar with SW-846, 40 CFR, and CLP protocols for data evaluation.

KARLA BIER

Semivolatiles GC Manager, Savannah Division

Ms. Bier has a B.S. degree in chemical engineering and two years experience in the analysis of environmental pollutants by gas chromatography. Prior to her association with Savannah Laboratories, she was a polymer chemistry research assistant at the University of Missouri-Rolla.

As manager of the semivolatile GC section of Savannah Laboratories, Ms. Bier has primary responsibility for overseeing the analysis of samples for pesticides, PCBs, herbicides, and other semivolatile organic compounds by GC. In addition, she is responsible for the implementation of new analytical procedures, training new personnel, and supervising maintenance and troubleshooting of semivolatile GC instrumentation. Her duties have given her extensive experience in SW-846, 40 CFR, and CLP protocols for data evaluation.

KATHERINE M. COOK

Semivolatiles GC/MS Chemist, Savannah Division

Ms. Cook has a B.S. degree in chemistry from the University of Georgia and four years of experience in the analysis of organic substances using HPLC, FTIR, GC, and GC/MS methods. Her background includes research and development for a pharmaceutical company, technical and chemical support for law enforcement agencies, and oceanographic research.

Ms. Cook began her career with Savannah Laboratories in 1990 as a GC/MS chemist. In this capacity, she is responsible for the operation and maintenance of two RTE/A GC/MS systems and determining the concentration of semivolatile organic compounds in extracts prepared from sample matrices using CLP, 8270, and 625 methodology.

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KENNETH R. AEGAN

Chemist, Savannah Division

Mr. Aegan has a B.S. degree in chemistry and an Associates degree in management and logistics from Georgia Southern University. He began his career with Savannah Laboratories in 1987. His work as both GC and GC/MS chemist has provided him with extensive knowledge of SW-846, 40 CFR, and CLP protocols.

HSIAO-LAN CHANG

Analyst, Savannah Division

Ms. Chang received a B.S. in horticulture in 1964 from National Taiwan University and an M.S. in plant sciences in 1972 from the University of Georgia.

From 1965 to 1968, Ms. Chang was a research assistant at Taiwan Agricultural Research Institute. From 1968 to 1969, she was laboratory technician for Naval Medical Research in Taipei.

Ms. Chang served as a laboratory technician at the U.S. Department of Agriculture's Stored Products Insects Research and Development Laboratory from 1980 to 1982 and again from 1987 to 1989.

Ms. Chang joined Savannah Laboratories in 1990 and is currently responsible for TOX determinations.

NANNETTE H. DASHER

Semivolatiles GC/MS Supervisor, Savannah Division

Ms. Dasher has a B.S. degree in chemistry with six years of laboratory experience. She joined Savannah Laboratories and Environmental Services, Inc. in 1985, and gained experience in sample preparation and determination of metals by inductively couples plasma (ICP) spectroscopy and organic sample preparation and analysis by IR and GC.

She has performed analyses by GC/MS for three years and is familiar with all SW-846 and CLP methodology.

In her current role, Ms. Dasher supervises the analysis of samples for semivolatile organic compounds. She is responsible for the training of new personnel for this section, implementing new analytical procedures, and supervising maintenance and troubleshooting of semivolatile GC/MS instrumentation.

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KARRI DERR

Volatiles GC/Manager, Savannah Division

Ms. Derr has a B.S. degree in animal science from Iowa State University. She was a research assistant for two years at Iowa State University Veterinary College, Large Animal Resources division.

She began her association with Savannah Laboratories in 1988, at which time she was responsible for performing determinations of metals utilizing inductively coupled plasma (ICP) emission spectroscopy and atomic absorption and flame spectroscopy.

Ms. Derr's current responsibilities include personnel, production, and quality control management of a GC/MS volatile group.

DAGMAR I. GOLEY

Inorganic CLP Coordinator, Savannah Division

Ms. Goley has an A.S. degree in chemical technology from Heinrich-Lanz-Schule II, in Mannheim, Germany. She began her association with Savannah Laboratories in 1987, and has gained experience in numerous inorganic preparation procedures.

Her duties at Savannah Laboratories include supervising sample preparation for all metals sample determinations and assisting in the computer generation of CLP forms for inorganic data packages. She is trained in total digestion, dissolved sample preparation, and inorganic extraction/concentration procedures as well as proper sample handling. She has been trained in clean room techniques and is familiar with EPA 600/4-79-020 SW-846 and CLP protocols.

SANDRA GROVENSTEIN

GC/MS Semivolatiles Supervisor, Savannah Division

Ms. Grovenstein received her B.S. degree in biology from Auburn University in 1978. She gained extensive experience in toxicology after graduation, working in several hospitals and laboratories. In 1986, she joined Laucks Testing Laboratories in Washington State where she was responsible for analysis and interpretation of GC/MS data relating to CLP 40 CFR and SW-846 protocols.

In 1990, Ms. Grovenstein joined Savannah Laboratories and is responsible for the supervision and analyses of samples and reporting of data for semivolatiles by GC/MS utilizing SW-846, 40 CFR and CLP protocol.

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SUSAN NORWOOD

ICP/AA Production Supervisor, Savannah Division

Ms. Norwood has a B.S. degree in chemistry from Armstrong State College and a B.A. degree in marketing from Georgia Southern College. She began work at Savannah Laboratories in 1988 in the inorganic section of the laboratory.

After working briefly in the determination of nutrients and general parameters, Ms. Norwood transferred to the metals section where she has worked extensively with both ICP, flame and furnace AA instrumentation. As well, she has been involved with sample preparation procedures for metals determinations. Ms. Norwood is currently responsible for supervision of ICP analyses, associated data handling, and maintenance and troubleshooting for this instrument. She has extensive experience with EPA 600/4-79-020, SW-846, and CLP protocols.

G. ANTHONY LOWMAN

Analyst, Savannah Division

Mr. Lowman has a B.S. degree in geology from Georgia Southern University. He has worked in the hydrology departments with both the U.S. Army Corps of Engineers and the Georgia Geological Survey and with the Skidaway Institute of Oceanography in Savannah, Georgia conducting research in coastal sedimentology.

Mr. Lowman joined Savannah Laboratories and Environmental Services, Inc. in 1989. He performs trace metal analyses of samples utilizing the ICP, Perkin Elmer HGA-400 Graphite Furnace and the SpectrAA-400 Zeeman Spectrophotometer as well as sample preparation by MIBK extraction and analysis, and back up for other trace metal work. He is familiar with EPA 600/4-79-020 and SW-846 and CLP protocols.

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PAUL E. MEYERS

Chemist/Corporate Safety Director, Savannah Division

Mr. Meyers obtained his B.S. in chemistry in 1953 from Marshall University. While still in school, he worked as a laboratory technician for Allied Chemical in South Point, Ohio. Within three years of his graduation from Marshall, he held the position of Senior Chemist with Allied.

From 1956 to 1969, he was successively plant superintendent, plant manager, and manager of manufacturing for Kaiser Agricultural Chemicals and Southern Nitrogen Company in Savannah, Georgia. His responsibilities at these companies included startup and operation of a fertilizer manufacturing complex. As manager of manufacturing, he was responsible for the manufacturing process at several locations.

From 1969 to 1970, he was Vice President of Valley Nitrogen Products in Fresno, California where he was responsible for startup and operation of what at that time was the largest fertilizer manufacturing complex west of the Mississippi River.

From 1970 to 1988, Mr. Meyers served as Vice President of System Services and Industrial Corporation where his responsibilities included plant maintenance, engineering, and consulting with clients.

In 1988, Mr. Meyers joined Savannah Laboratories. His initial responsibilities provided him with experience in sample preparation for metals determinations and ICP operation. He has worked with and supervised extraction, preconcentration, and other sample preparation techniques for dioxin samples. Mr. Meyers currently is responsible for nutrient analysis employing the TRAACS autoanalyzer system as well as acting Corporate Safety Director for Savannah Laboratories.

RUTH D. RANKIN

Digest Supervisor, Savannah Division

Ms. Rankin has a B.S. degree in biology. She has five years laboratory experience with Southeast Labs in Atlanta, Georgia, where she performed various general chemistry determinations, sample digestions, and metals and mercury determinations by atomic absorption spectroscopy.

Ms. Rankin began her career with Savannah Laboratories in 1987. Her duties include supervising the atomic absorption section of the laboratory. She is responsible for organizing the sample load, recording results on the worksheets, and performing analyses using the Perkin Elmer, Jarrell Ash, and Varian atomic absorption spectrophotometers. Her work has provided her with an extensive knowledge of EPA 600.4-79-020, SW-846, and CLP protocols.

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J. ROBERT PADDISON, JR.

Volatiles GC/MS Supervisor, Savannah Division

Mr. Paddison has a B.S. degree in chemistry from the Georgia Institute of Technology and a M.S. degree in biochemistry from the University of Wisconsin - Madison. He began his association with Savannah Laboratories in 1990.

Mr. Paddison started his career at the University of Wisconsin Clinical Cancer Center isolating and quantitating amino biphenyl-DNA adducts using HPLC, LC, and scintillation counting. Most recently, he has worked as an analyst to perform isomer specific quantitation of PCBs in sediment samples from Green Bay, Wisconsin, using GC and GC/MS techniques. This work was conducted in the Water Chemistry Program, U.S. - Madison, as part of the EPA-directed Green Bay Mass Balance Study.

Mr. Paddison's current responsibilities at Savannah Laboratories are supervision of a GC/MS volatiles section utilizing 40 CFR, SW-846, and CLP protocols.

MICHAEL J. SALUM

Organic Extraction Manager, Savannah Division

Mr. Salum has a B.S. degree in agronomy with a specialization in soil science from the University of Georgia. Mr. Salum spent over two years as an on-site contractor at the U.S. EPA Region IV Laboratory in Athens, Georgia, performing and supervising semivolatile organic extractions. He was also responsible for the preparation of blind QA samples for all PRP and CLP laboratories in Region IV. His background includes research in soil conversation and soil chemistry at the University of Georgia.

Mr. Salum began his association with Savannah Laboratories in 1990 as a GC pesticide residue chemist. He is currently responsible for the operation of the organic extraction/GC screening laboratory. His duties include personnel, production, and quality control management of the extraction lab for all organic parameters.

ANGELA F. STEWART

Chemist, Savannah Division

Ms. Stewart has a B.S. degree in chemistry from Armstrong State College. She joined Savannah Laboratories in 1989. She initially worked in the extraction and concentration of samples for semivolatile GC/MS analysis.

Ms. Stewart is currently responsible for the determination of semivolatile organic compounds by GC/MS. Her work has provided her with extensive experience employing SW-846, 40 CFR, and CLP protocols.

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ANGELA M. WEIMERSKIRK

General Laboratory Supervisor, Savannah Division

Ms. Weimerskirk has a B.S. degree in chemistry. She began her career with Savannah Laboratories in 1986, at which time she was responsible for the determination of trace metals by ICP and the determination of mercury by cold vapor AAS. She has extensive experience in ion selective electrode determinations of fluoride, ammonia, and TKN, and several years experience with the determination of ions utilizing ion chromatography.

Ms. Weimerskirk is responsible for the organization, coordination, and operation of the Traacs 800 autoanalyzer, the ion chromatograph, and ion selective electrode instrumentation section of the laboratory. She has been involved in method development of cyanide and phenolics by autodistillation/autoanalysis. In addition, her work has improved the efficiency and accuracy of sulfide determinations. She is familiar with EPA 600/4-79-020, SW-846, and CLP protocols.

ANGELA ZEALY

Chemist, Savannah Division

Ms. Zealy has a B.S. degree from Armstrong State College with a major in biology and a minor in chemistry. She joined Savannah Laboratories in 1988.

Ms. Zealy began her tenure with Savannah Laboratories performing microbiological determinations. Since that time, she has gained extensive experience in the extraction, clean-up, dilution, and preconcentration of samples for the determination of pesticides, herbicides, and PCBs. Her current position is GC chemist in the semivolatiles section of Savannah Laboratories.

Ms. Zealy's responsibilities include GC/FID determination of phenolic compounds, phthalate esters, and PAH compounds. In addition, she is experienced in petroleum product identifications. Her work has given her experience with both SW-846 and 40 CFR methodologies.

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THOMAS L. STEPHENS

Vice President/Project Manager, Tallahassee Division

Mr. Stephens has a B.S. degree in chemistry and 20 years experience in analyses of pollutants in groundwater, sediments, tissues, agricultural products, wastewater, drinking water and hazardous wastes utilizing GC, GC/MS and HPLC analytical techniques. Prior to his association with Savannah Laboratories in 1985, he was supervisor of the organic section of the Florida Department of Environmental Regulation (DER) laboratory in Tallahassee and supervisor of the Florida Department of Agriculture Pesticide Residue Laboratory. In these capacities, he has gained an enormous amount of experience in laboratory management, quality assurance, method development, mass spectrometry interpretation and verification of results. He has attended several regulatory training schools and presented technical presentationsa at several pesticide residue conferences and regional meetings in the southeast.

Mr. Stephens is thoroughly familiar with DER specific regulatory sampling procedures, analytical quality assurance and reporting requirements for hazardous and solid waste, air quality, groundwater, wastewater, and drinking water. His technical background enables him to provide accurate and cost effective assistance for the environmental and regulatory needs of Florida clients. He is a member of the Florida Society of Environmental Analysts, the Florida Association of Environmental Professionals, the Florida Environmental Auditors Association, and the Florida Air and Waste Management Association.

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JANET PRUITT

Laboratory Director/Project Manager, Tallahassee Division

Ms. Pruitt has a B.S. degree in chemistry and a Master of Public Health degree. Her 25 years experience in the environmental field began with the South Carolina Department of Health and Environmental Control (SCDHEC) where she supervised the Environmental Chemistry Section. After nearly thirteen years with SCDHEC, she joined the United States Geological Survey (USGS) National Water Quality Laboratory in Atlanta, Georgia.

At the USGS laboratory, she was responsible for the analyses of water, sediment, and fish tissues for various organic parameters utilizing gas chromatography, mass spectrometry and computer data systems. She also served as quality assurance officer for the organic chemistry section.

In 1984, Ms. Pruitt transferred to the Tallahassee office of USGS where she was responsible for appraising water resources and providing basic hydrologic data on both surface and ground water in Florida. She was project chief of three investigative hydrologic studies, assisted with three water quality studies, and coauthored an indexing and classification system for earth-science data bases. She also served as technical advisor in the field of analytical organic chemistry for district, regional and headquarters personnel in the Water Resources Division.

Ms. Pruitt joined Savannah Laboratories in 1987 as laboratory manager/QA manager. In her current position as laboratory director/project manager, she is responsible for the operation of the Tallahassee Division, and provides clients with assistance in field and analytical requirements.

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WAYNE WORD

Project Manager, Tallahassee Division

Mr. Word has a B.S. degree in chemistry and 16 years experience in the environmental field. He began his career with Technical Services, Inc. (TSI) in Jacksonville, Florida, where he was organics department manager responsible for general analysis and performing and instituting instrumental methods for new clients. After 10 years with TSI, Mr. Word joined OHM Corporation where he began as a project chemist and worked his way up to laboratory manager within four years.

Mr. Word has experience in most major analytical techniques including: GC, GC/MS, HPLC, FTIR, UV/VIS, TOC, TOX, ICP, AA, and manual and automated potentiometric and spectrophotometric methods. He also has extensive experience with SW-846 and EPA approved methods. In addition, Mr. Word has received specialized training from Hewlett-Packard, Varian Associates, Jarrell-Ash, Perkin Elmer, and the American Chemical Society on instrumental and management techniques.

Mr. Word joined Savannah Laboratories as project manager. In this position, he is able to provide technical assistance and support to clients for field and analytical services.

HENRY BEAUCHAMP

Laboratory Manager, Tallahassee Division

Mr. Beauchamp holds a B.S. degree in chemistry from the University of Florida and has completed postgraduate course work in biochemistry. Prior to his association with Savannah Laboratories in 1989, he worked for the Florida Department of Agriculture and the University of Florida in biochemical laboratory analysis.

Mr. Beauchamp has supervised all volatile organic compound determinations by GC and GC/MS, and has been actively involved in GC/MS analysis, spectral interpretation, data reporting, and instrument maintenance.

In his current role at Savannah Laboratories, Mr. Beauchamp is responsible for the management of technical personnel, overseeing all method development and adherence to the Comprehensive QA Plan, coordinating all analyses with section managers, as well as handling all requests for laboratory supplies and instrument repairs.

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ELIZABETH SCHNEIDER

Quality Assurance Hanager, Tallahassee Division

Ms. Schneider has a B.S. degree in biology and over 17 years experience with gas chromatography and high performance liquid chromatography analyses. At Savannah Laboratories, Ms. Schneider is responsible for ensuring that method QA requirements are met, issuing and evaluating inhouse check samples, and analyst training in safety, QA procedures, and analytical methodology. Ms. Schneider has a thorough knowledge of QA requirements, procedures, and evaluation as they apply to EPA-approved methodology. She is responsible for research into new methodologies and has developed several procedures now in use at Savannah Laboratories. She also supervises analyses of ordinance and explosives according to USATHAMA methods.

Prior to her association with Savannah Laboratories in 1987, Ms. Schneider was lead technician in the quality assurance department for the Olin Corporation. There she supervised a staff of professionals in areas such as chemical, raw materials, water and waste treatment, ballistics and instrumental training. She is highly skilled in the operation and repair of gas chromatographs and high performance liquid chromatography systems.

DANA TILL

Chemist, Tallahassee Division

Ms. Till holds a B.S. degree in chemistry from Pembroke (NC) State University. Prior to coming to Savannah Laboratories, she was employed by the City of Raeford and Berry College.

During her employment with the City of Raeford, Ms. Till was responsible for the supervision of all daily laboratory activities, training of laboratory personnel, purchasing of equipment for the plant and laboratory, and performing wastewater analysis. She established a quality assurance program to test laboratory procedures, techniques, and methodology. She also was responsible for bringing the wastewater treatment laboratory into compliance with the State of North Carolina's standards, as well as publishing an operations manual for the laboratory.

Her current responsibilities at Savannah Laboratories include the analysis of samples for polynuclear aromatic hydrocarbons and pesticides by GC.

BERNARD ASH

Chemist, Tallahassee Division

Mr. Ash has a B.S. degree in chemistry from Florida A&M University and has completed Medical Laboratory Specialist Training in the U.S. Air Force. Prior to joining Savannah Laboratories, he was employed as a medical technologist in the Air Force as well as with Gadsden Memorial Hospital and Tallahassee Memorial Regional Medical Center.

Mr. Ash's duties at Savannah Laboratories include performing analyses of metals in environmental matrices employing ICP and AA furnace techniques.

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TODD BAUMGARTNER

Inorganic Section Manager, Tallahassee Division

Mr. Baumgartner holds a B.S. degree in chemistry and has completed one year of postgraduate course work. He began his career with Savannah Laboratories in 1985. His training since joining Savannah Laboratories has been broad, encompassing sampling as well as nutrient, volatile, organic compounds, and metal determinations.

Mr. Baumgartner's current duties as manager of the inorganic section include overseeing all aspects of analysis from digestion to data reporting for metals, general (wet chemistry), and bacteriological parameters. He is responsible for ensuring adherence to QC procedures and method requirements, instrument troubleshooting and maintenance, and ordering of lab supplies. He is especially experienced in metal determination by ICP, furnace AA, and cold vapor AA.

ROBERT D. DRIVER

Analyst, Tallahassee Division

Mr. Driver has a B.S. degree in business administration and two years experience as a laboratory analyst. He has experience over a wide variety of chemical analyses including general chemistry, bacteriology, extractions, metals, IR, and digestion.

Currently, Mr. Driver's primary responsibility is HPLC analyses of pesticides and related compounds.

D. WAYNE HIGGINS

Analyst, Tallahassee Division

Mr. Higgins has a B.S. degree in nutritional science with a minor in chemistry from Florida State University. Prior to coming to Savannah Laboratories, he was employed by Florida State University Chemistry Department.

Mr. Higgin's duties include the analysis of samples for metals by ICP and furnace AA methodologies. He also checks data entered on worksheets for his section making sure QA/QC requirements are met and all data is entered correctly.

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DAVID KARNS

Semivolatiles Organic Manager, Tallahassee Division

Mr. Karns holds a B.S. degree in chemistry from the University of South Florida. His experience includes analysis of semivolatile organic compounds by GC/MS, spectral interpretation, and adherence to SW-846, 40 CFR, and CLP methodologies.

Mr. Karns' has extensive knowledge of semivolatile method requirements, data reporting, and QC requirements. His duties as manager consist of installing new software updates on Hewlett-Packard GC/MS systems, advising and training on all systems, scheduling workloads, maintaining and troubleshooting instruments, and providing technical information to the laboratory director regarding new instrumentation and method requirements. Mr. Karns is experienced in review of CLP data packages.

JOSEPH B. NORTH

Analyst, Tallahassee Division

Mr. North holds a B.S. degree in biology from Florida State University. Prior to his employment with Savannah Laboratories, he worked for Environmental Planning and Analysis, Inc. as a laboratory technician.

Mr. North's duties include the analysis of samples for biological parameters, including BOD, COD, bacteria, and other biological laboratory duties.

TIMOTHY PRESTON

Chemist, Tallahassee Division

Mr. Preston holds a B.S. degree in chemistry with a minor in mathematics from Florida State University and also has an AA degree in liberal arts from Miami-Dade Community College. He is a member of Alpha Chi Sigma Chemistry fraternity.

Mr. Preston is a GC/MS chemist and is responsible for analyzing environmental samples for volatile organic compounds using packed and capillary column GC/MS. He is also responsible for the review and interpretation of spectral data and review and analysis of CLP data packages.

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PAUL RYGIEL

HPLC/IR Manager, Tallahassee Division

Mr. Rygiel has a B.A. degree in biochemistry from Florida State University. Prior to joining Savannah Laboratories, he was employed as a chemist at the Florida Department of Business Regulation where he gained two years experience in gas and liquid chromatography.

Mr Rygiel's current responsibilities include overseeing the analysis of soil and water samples by EPA methods 632, 531, 8320, and other methods for explosives, formaldehyde, and water soluble pesticides and herbicides by HPLC, and the IR analysis of soil and water samples for oil and grease and total petroleum hydrocarbons.

DEBORAH SHERWIN

Biological/General Lab Supervisor, Tallahassee Division

Ms. Sherwin is a graduate of Florida State University where she obtained a B.S. degree in biological science. Her educational background includes completion of the Liberal Studies Honors program and memberships in Phi Eta Sigma National Honor Society and Golden Key National Honor Society.

Ms. Sherwin joined Savannah Laboratories as an analyst in the Biological/General Lab section. Her current duties as supervisor include analysis and supervision in all areas of bacteriology and general chemistry. She is involved in analyses of coliforms, titrations, BODs, solids determinations, and determinations of physical parameters using EPA approved methodologies.

RICHARD A. STEPHENS

Volatiles Organics Manager, Tallahassee Division

Mr. Stephens holds a B.S. degree and Postgraduate Certificate of Education from the University of Wales. His studies were in biology and zoology, and prior to his employment at Savannah Laboratories, he was a science educator.

Mr. Stephens' duties at Savannah Laboratories include supervision of analyses by GC and GC/MS of volatile organic compounds, data interpretation and reporting, instrument maintenance and troubleshooting, review of CLP data packages, and ordering of gases and lab supplies for the volatiles section.

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MARTIN THOMAS

Technician, Tallahassee Division

Mr. Thomas came to Savannah Laboratories with more than 16 years experience. His duties at Savannah Laboratories have included supervision of the extraction laboratory as well as performance of extractions for all organic parameters. He has also been responsible for analysis of samples for petroleum hydrocarbons by IR techniques. At the present time, he analyzes all water samples for EDB following EPA methodology.

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JESSE L. SMITH

Laboratory Director/Project Manager, Mobile Division

Mr. Smith has a B.S. degree and has successfully completed the majority of graduate course work in a masters program in chemistry.

Mr. Smith is the laboratory director of Savannah Laboratories, Mobile, Alabama Division and is responsible for overall management of this laboratory. He supervises project managers, QA and department managers, and ensures departments have adequate laboratory equipment and personnel to perform their jobs. He provides oversight in developing new laboratory methods and analytical techniques to meet client needs as regulatory programs change.

Mr. Smith also functions as project manager and is the primary contact for his clients. He is responsible for assisting clients with pre-sampling discussions, suggesting analytical approaches, meeting regulatory agency requirements, and developing special analytical and treatability techniques. He is thoroughly familiar with EPA methods and QA/QC requirements for RCRA, NPDES, SDWA, and other EPA regulatory programs.

Mr. Smith is an active member of the American Chemical Society and has attended ACS-sponsored courses in gas chromatography and atomic absorption spectroscopy. He has attended numerous continuing education courses covering new EPA regulatory programs.

Mr. Smith joined Savannah Laboratories and Environmental Services, Inc. in 1987, as a project manager and was promoted to laboratory director of the Mobile, Alabama Division when it opened in 1988. Prior to joining Savannah Laboratories, he had fifteen years consulting laboratory experience as a bench chemist, supervisor, and laboratory manager for Southeast Laboratories, Inc. in Atlanta, Georgia. He has extensive experience in gas chromatography analysis of pesticide residue/PCBs and volatiles, metals by atomic absorption spectroscopy including graphite furnace analyses, and general chemistry laboratory experience. He has a broad base of experience in a variety of analytical chemistry and microbiological methods including: EPA, ASTM, NIOSH, AOAC, FDA, and USDA.

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J. MICHAEL NANCE

Project Manager, Mobile Division

Mr. Nance has a B.S. degree from the University of North Alabama and an M.S. degree in environmental science from the University of South Alabama.

Mr. Nance's primary responsibility with Savannah Laboratories is project management of permitted wastewater and drinking water and RCRA projects as required by EPA and state regulatory programs. Mr. Nance provides oversight and supervises the field activities of the Mobile Division sampling team. He is highly skilled at coordinating and conducting field investigations.

Mr. Nance has kept current with changes and refinements in existing regulatory programs through continuing education courses in Hazardous Waste/Land Disposal Bans, Reauthorized RCRA, Superfund (SARA), and NPDES storm water regulations. He has completed the forty-hour "Hazardous Assessment and Response Management" training program (20 CFR 1910.120). This certification is kept current through annual update courses.

Mr. Nance has seventeen years experience in a wide variety of industrial laboratory and field investigations. Project experience includes: hazardous waste characterization, sediment and water quality studies, remedial investigations, and groundwater monitoring projects.

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MICHELE H. LERSCH

Laboratory QA Manager, Mobile Division

Ms. Lersch has a B.S. degree in medical technology with concentrations in chemistry and microbiology from the University of South Alabama. She has taken postgraduate courses in management and business law.

Ms. Lersch is responsible for managing the quality assurance program for the Mobile Division laboratory. She initiates certifications with state and federal agencies, renews certifications, conducts internal audits, works with external auditors, edits QA plans and SOPs, and reviews project files. Her duties include initiating and maintaining training files for the technical staff and maintaining technical information resources and methods.

Prior to her employment with Savannah Laboratories, Ms. Lersch was employed as metals analyst with Thompson Engineering Testing. She specialized in trace metals determinations using flame and graphite furnace AAS techniques. She also became experienced in inorganic analyses using wet chemical methods.

When Ms. Lersch joined Savannah Laboratories in 1988, she was promoted to Inorganic Laboratory Manager. She gained extensive experience using inductively coupled plasma (ICP) techniques for metals determinations. She was responsible for instrument maintenance and trouble-shooting as well as day-to-day implementation of QC for the inorganic section. She became thoroughly familiar with EPA methods and reporting and QC requirements for environmental samples requiring metals and wet chemistry analyses.

Ms. Lersch has ten years of laboratory experience, seven of which have been directly related to the chemical and biological analysis of water, wastewater, soils/sediments, and hazardous waste samples. She is familiar with SW-846 and 40 CFR methods, protocols, and QC requirements for both organic and inorganic analysis.

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VAN PHAM

Laboratory Manager, Mobile Division

Dr. Pham has a Ph.D. in organic chemistry from Georgia Institute of Technology and a B.S. degree in chemistry from Saigon University.

Dr. Pham joined Savannah Laboratories in 1990 as a GC/MS chemist. She was responsible for the analysis of base/neutral and acid semivolatile organics in environmental samples. In 1991, Dr. Pham was promoted to department manager and QC coordinator of the organic and extraction sections and to laboratory manager in 1992. She is responsible for the organic analytical method development, supervising, training new personnel, and maintaining and troubleshooting the GC/MS, GC, IR, ICP and AA/GF instruments.

Dr. Pham is an active member of the American Chemical Society. She has attended several ACS-sponsored seminars on a variety of topics including GC and GC/MS techniques and methods. She took the quality assurance course offered by the EPA as part of its Seventh Annual Waste Testing and Quality Assurance Symposium held in Washington, D.C.

Prior to joining Savannah Laboratories, Dr. Pham was employed with Eagle-Picher Environmental Service. She performed a variety of organic and air pollutant analyses for volatile compounds, pesticides, herbicides, PCBs, PAHs, phenols, and dioxins. From 1987 to 1989, Dr. Pham was a postdoctoral associate with the University of Georgia, Department of Chemistry. She worked on enzymatic synthesis and reaction, and authored and co-authored a number of publications in the Journal of the American Chemical Society.

Dr. Pham has extensive knowledge of EPA SW-846, 40 CFR, NIOSH, and ASTM methods and CLP protocols. She has several years of experience working with GC, GC/MS, FT-NMR, UV-VIS spectroscopy, and HPLC instruments. She is familiar with several instrument data systems including PE-Nelson, Maxima 280, and ChemStation. She is well-versed in the use of Lotus 1-2-3 and Quatropro as part of the organic lab information management systems.

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CORA M. PATE

Volatiles Manager, Mobile Division

Ms. Pate has a B.S. degree in biology and a minor in chemistry from the University of South Alabama.

Ms. Pate is responsible for supervising the volatiles laboratory. Her duties include training analysts, instrument maintenance and data review as well as the analysis of environmental samples for volatile organic compounds by GC/MS according to EPA methods 624 and 8240. Ms. Pate has previously analyzed volatile organic compounds by gas chromatography using Hall, Photo Ionization (PID), and Flame Ionization (FID) detectors according to EPA methods 501.1, 502.2, 601/602, and SW-846 8010/8020. She interprets chromatograms, calculates and reports results and checks QC on these instruments.

Ms. Pate has attended seminars and courses on gas chromatography involving column maintenance and operation according to new methods and techniques.

Ms. Pate previously worked as an office manager before obtaining her degree. Her duties included training and maintaining the office staff as well as being a personal assistant to the company president. She was responsible for implementing new computer programs and updating and efficiently utilizing existing programs and hardware.

Ms. Pate has three years experience in an analytical and consulting laboratory atmosphere with two years of this in a supervisory position. She is familiar with SW-846 and 40 CFR methods, protocols, and QC requirements for the volatiles GC section.

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BRUCE H. BARRETT

Chemist, Mobile Division

Mr. Barrett has a B.S. degree in chemistry from Wittenberg University in Springfield, Ohio. He has done biochemical research of photophosphorylaton mechanisms in single celled algae at Kettering Memorial Laboratories in Yellow Springs, Ohio.

Mr. Barrett joined Savannah Laboratories in 1990 as a chemist in the general laboratory. He is the senior level chemist for this department and performs cyanide, nutrient, titrimetric and demand analyses. Mr. Barrett possesses considerable problem solving skills for troubleshooting methods and dealing with difficult sample matrices. He is well versed in computer programming and practical computer applications.

Mr. Barrett previously worked as a research lab technician with the Institute of Paper Chemistry studying reaction experiments on terpene hydrocarbons using gas-liquid chromatography and IR and NMR. He worked four years as senior chemist for Ventron Corporation troubleshooting products. He spent four years working for Lincoln Pulp and Paper as technical director and six years in the same capacity at Boise Cascade Corporation. As technical director, he was responsible for environmental and water plant operations as well as quality control of pulp and paper.

Mr. Barrett has twenty-five years of analytical experience in industrial, environmental, and chemical analyses. He is thoroughly familiar with EPA, ASTM, TAPPI, and NIOSH methods for wet chemical testing of a variety of sample matrices including: waters, wastewaters, soils/sediments, and hazardous waste.

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REBECCA BOWEN

Analyst, Chemical Hygiene Officer, Mobile Division

Dr. Bowen received a B.S. degree in biology in 1979 and a Ph.D. degree in basic medical sciences in 1989 from the University of South Alabama.

As the Chemical Hygiene Officer, Dr. Bowen is responsible for training and educating employees about safety rules and regulations. She enforces the company safety policies and ensures that the laboratory chemical hygiene plan complies with OSHA guidelines. Additionally, Dr. Bowen is the primary analyst for determining pesticide residues, herbicides, and PCBs in extracted samples using GC-ECD techniques.

Prior to employment with Savannah Laboratories, Dr. Bowen performed post-doctoral research at MacMaster University in Hamilton, Ontario. Her research involved investigating the pharmacological influences of prostaglandin and cyclic nucleotide metabolism on blood platelets in human tumor cells. She has several publications relating to her research in medical and other scientific journals.

Dr. Bowen has one year of environmental laboratory experience. She is familiar with SW-846, 40 CFR Part 136, and SDWA protocols for the parameters for which she is responsible.

PANDA CARTER

Analyst, Mobile Division

Ms. Carter has a B.S. degree in biology with a minor in chemistry from the University of South Alabama.

Ms. Carter joined Savannah Laboratories in 1991. Her primary responsibilities include sample analyses by ICP, flame, GFAA, and cold vapor/hydride generation atomic absorption techniques.

Ms. Carter has 12 years experience in atomic absorption spectroscopy and various wet chemistry techniques from past employment with Union Carbide Corporation and Protein Technologies International. She is thoroughly familiar with EPA-600 and SW-846 sample preparation and analytical methods for drinking waters, wastewaters, soils/sediments, hazardous waste samples, and TCLP extracts as well as the associated QC requirement.

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CHRISTOPHER A. COOK

Field Manager, Mobile Division

Mr. Cook received a B.A. degree in biology from Clemson University in May, 1990. His primary responsibilities with Savannah Laboratories is scheduling field activities, coordinating field logistics for environmental programs and the field team leader for sampling events.

Mr. Cook joined Savannah Laboratories in 1990, and has developed operating procedures which have satisfied the scrutiny of federal and state regulatory agencies performing on-site audits. Mr. Cook is skilled in providing field support for water quality programs, groundwater monitoring, solid/hazardous waste characterization, air monitoring for personnel exposure and other environmental programs.

Mr. Cook has completed the forty hours "Hazardous Assessment and Response Management" training course to meet the requirements of 20 CFR 1910.120.

CEDRIC CRAWLEY

Technician/Extraction Lab Group Leader, Mobile Division

Mr. Crawley has completed course work toward a degree in computer science. As extractions lab group leader, Mr. Crawley is responsible for scheduling the organic extractions of BNAs, herbicides, pesticides, PCBs, phenols, phthalates, and PAHs from drinking waters, wastewaters, solids/sediments, and TCLP extracts for analysis by GC and GC/MS. He performs analysis of oil and grease and petroleum hydrocarbons by gravimetric and infrared techniques, and total phenolics and MBAS using organic extraction/spectrophotometric techniques.

Mr. Crawley's previous laboratory experience includes field sampling and analysis. He was responsible for sampling of NPDES projects and analysis of such parameters as D.O., conductivity, residual chlorine, turbidity, and pH.

Mr. Crawley has three years of environmental laboratory experience, one of which has been in a group leader role. He is familiar with SW-846 and 40 CFR Part 136 methods, protocols, and associated QC requirements for semivolatile organic extractions.

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SHERYL S. FULLER

Chemist, Mobile Division

Ms. Fuller has a B.S. degree in chemistry from Stillman College. She has three years of analytical laboratory experience in the analysis of nutrients in waters, wastewaters, soils and sediment samples. She is also experienced in teh analysis of cyanides in a variety of sample matrices.

Since joining Savannah Laboratories in 1989, Ms. Fuller has gained extensive experience using wet chemical and spectrophotometric methods and ion specific electrodes. She is currently specializing in the determination of demand analyses and phosphorous in waters, wastewaters, soils and sediments. Ms. Fuller is thoroughly familiar with EPA methodologies for wet chemistry testing and the associated QC requirements.

STEPHANIE H. JONES

Field Scientist, Mobile Division

Ms. Jones has a B.S. degree in geology from the University of South Alabama. She joined Savannah Laboratories' field sampling department in 1991 as a trainee, bringing with her an extensive background in field sampling and analytical techniques.

Ms. Jones has been involved in several major sampling projects for drinking water, groundwater, stormwater, soil/sediment and hazardous waste samples. She has worked with clients in establishing sampling programs and writing SOPs.

Ms. Jones is thoroughly familiar with SW-846 sampling protocols, and is able to apply her extensive practical experience to any special sampling techniques as required.

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SHAU-WEI LI

Chemist, Mobile Division

Dr. Li received her Ph.D. in 1989 and M.S. in 1986 in organic chemistry from Shanghai Institute of Organic Chemistry, Shanghai, China. She received her B.S. degree in chemistry from the Zhe-Tsian University in 1983.

Dr. Li joined Savannah Laboratories in 1991 as a pesticide residue chemist in the semivolatile gas chromatography (GC) laboratory. She is responsible for the analysis of pesticides/PCBs, PAHs, phenols and microextractable compounds in environmental samples. she is familiar with the operation of GCs equipped with electron capture, flame ionization, nitrogen-phosphorous detectors using capillary and packed columns. she is familiar with the Nelson and Hewlett Packard computerized data management system to collect and process analytical data.

Dr. Li has attended seminars and courses on gas chromatography involving column maintenance and operation according to new methods and techniques. She regularly attends the Gulf Coast Chromatographers Discussion Group meetings.

Prior to joining Savannah Laboratories, Dr. Li was a post-doctoral research associate for one year with the University of South Alabama Chemistry Department. she conducted research in the chemical synthesis of naturally occurring antibiotics.

Dr. Li is thoroughly familiar with the GC methods regarding sample preparation and analytical methods for drinking water, wastewater, soils/sediments, hazardous waste samples and TCLP extract analyses. she has extensive knowledge of EPA SW-846, 40 CFR Part 136 protocols and associated QC requirements.

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KATHERINE WYNN MORGAN

Sample Coordinator, Mobile Division

Ms. Morgan has completed several courses toward a B.S. degree in chemistry. She is currently enrolled at the Faulkner State University and working toward completion of that degree.

Ms. Morgan's duties at Savannah Laboratories include filling client requests for sample bottles, resolving discrepancies in requests for analysis at sample receipt, and disposing of samples when analysis and reporting are complete. She coordinates all project orders from sample login to computer login and interfaces with the clients and project managers to facilitate the sample flow through the laboratory. In addition, she is responsible for ordering and receiving supplies and materials for the laboratory.

Prior to her employment with Savannah Laboratories in 1988, she worked in the petrochemical industry. As a laboratory technician, she performed chemical analysis of petrochemical products. She also developed skills such as data recording on a computer data system, interacting with clients, packaging and mailing restricted articles, and purchasing/receiving for the laboratory.

Ms. Morgan has eight years of laboratory bench experience. She has extensive knowledge of field sampling procedures and packaging and transporting requirements of a variety of chemicals. Ms. Morgan is familiar with the laboratory information management system of Savannah Laboratories. She is available to assist clients with scheduling bottle order shipments and receipt of samples in the laboratory.

EDWARD OETKEN

Metals Digestion Coordinator, Mobile Division

Mr. Oetken has a B.S. degree in biomedical sciences with a chemistry minor from the University of South Alabama.

At Savannah Laboratories, Mr. Oetken supervises all digestion procedures and sample receipts in the metals section. In this role, he has acquired knowledge of laboratory techniques and become familiar with SW-846 and EPA 600 protocols.

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TRACY S. OWENS

Metals Supervisor, Mobile Division

Ms. Owens has a B.S. degree in environmental sciences from Troy State University. She joined Savannah Laboratories in 1990, and has specialized in the determination of trace metals analysis using inductively coupled plasma (ICP), flame, cold vapor, hydride generation and graphite furnace atomic absorption spectroscopy techniques.

Ms. Owens was promoted to metals lab supervisor in 1992. she is responsible for scheduling digestion and analysis for drinking water, waste water, soil/sediment, hazardous waste and TCLP projects. she ensures that proper preparation and analysis techniques are followed and method QC requirements are met. Ms. Owens oversees instrument maintenance and troubleshooting for the metals department.

Ms. Owens has three years experience in the laboratory. She is familiar with EPA SW-846 and Title 40 Part 136 test method and associated QC requirements.

SONYA REYNOLDS

Inorganics Group Leader, Mobile Division

Ms. Reynolds has a B.S. degree in biology and environmental science from Livingston University. She was a founding member of the L.U. Conservacy group and is active in promoting environmental awareness.

Since joining Savannah Laboratories in 1991, she has gained extensive knowledge of EPA methodologies for demand, nutrient, and general wet chemical analyses. She is familiar with SW-846, Standard Methods, and EPA 600 series methods of analysis for waters, wastewater, soil/sediment, and hazardous waste samples as well as the associated QC requirements.

Ms. Reynolds is responsible for analysis of TOC and COD, coliforms by both membrane filtration and multiple tube techniques, sulfides, and chlorides. She also oversees the analytical work of several other analysts and troubleshoots problems with data and QC.

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LETITIA SAUNDERS

Analyst, Mobile Division

Ms. Saunders has a B.A. degree in biology from Berea College. She has completed two years of study toward a degree in dentistry at the University of Kentucky. She joined Savannah Laboratories in 1991 as a nutrients analyst. In this position, she was responsible for the determination of TKN, NH $_3$, NO $_3$, and NO $_2$ in waters, wastewaters, soils, and sediments. She is familiar with EPA-600 wet chemistry methodologies and associated QC requirements.

Ms. Saunders has moved to the volatiles laboratory and is responsible for the analysis of environmental samples for volatile organic compounds by gas chromatography using Hall and Photo Ionization (PID) detectors according to EPA mehtods 501.1, 502.2, 601/602, and SW-846 8010/8020. She interprets chromatograms, calculates and reports results, and checks QC for these instruments.

Prior to joining Savannah Laboratories, Ms. Saunders was a research technologist at the University of South Alabama. She is experienced in the use of a variety of spectrophotometric equipment and is familiar with HPLC and column chromatography techniques.

NAN SCARBROUGH

Analyst, Mobile Division

Ms. Scarbrough received a B.S. degree in wildlife biology from Livingston University. She also holds a Master of Education degree from Livingston University. Her primary responsibility at Savannah Laboratories is analyses of cyanide in waters, wastewaters, soils/sediments, and hazardous waste samples.

Prior to joining Savannah Laboratories in 1991, Ms. Scarbrough worked as a lab technician for the Alabama Co-op Fish and Wildlife Research Unit performing habitat mapping and collection and identification of fish. She also has experience in bioassay analysis from her employment as an aquaculture specialist with TAI Environmental Sciences, Inc.

Ms. Scarbrough has two years of laboratory experience. She is familiar with SW-846 and EPA-600 methods for cyanide analyses on a variety of sample matrices.

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JOHN SIMS

Analyst, Mobile Division

Mr. Sims has a B.S. degree in physics with a minor in computer science from Alabama State University. He joined Savannah Laboratories in 1991 as an analyst with the responsibility of extracting both volatile and non-volatile TCLP samples.

Mr. Sims is currently responsible for the analysis of semivolatile extractable organics, primarily phenols, PAHs, and phthalate esters, by GC-FID methods. He is trained in both 40 CFR and SW-846 methods and QC requirements.

Prior to joining Savannah Laboratories, Mr. Sims worked as a lab technician in the biomedical research department at Alabama State University. He also co-authored four publications regarding PIXIE analysis techniques for analyzing environmental samples. He has also worked as a computer programmer with Computer Graphic Management, writing programs on level 6 machines for report formats, payrolls, and invoices for a variety of businesses.

RHODA SMITH

Office Manger/Data Coordinator, Mobile Division

Ms. Smith attended Georgia State University in Atlanta, Georgia, where she majored in business administration. She has held various positions in the financial field. She joined Savannah Laboratories in 1988 as office manager. Her duties include supervision of office personnel and coordination of data and billing. She is thoroughly familiar with Savannah Laboratories' Information Management System (LIMS) and is responsible for editing reports for complex projects. She reports directly to the Laboratory Director and works with project mangers, analysts, and clients to coordinate prompt data flow through the laboratory.

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VIRGINIA VASQUEZ

Analyst, Mobile Division

Ms. Vasquez has a B.S. degree in chemistry/biology from the University of Guadalajara, Mexico. She has four years of analytical laboratory experience. She is familiar with EPA SW-846 and CFR Title 40 Part 136 test methods, protocols, and associated QC requirements. She was previously employed as a laboratory supervisor in a wastewater control laboratory. She was responsible for assuring that proper methods, techniques and procedures were used. She also worked as an analyst in an instrumental, wet chemistry and biology laboratory.

Ms. Vasquez's primary responsibility at Savannah Laboratories is the analysis of environmental samples for volatile organic compounds by gas chromatography using Hall and flame ionization detectors (FID) according to EPA Methods 601/602 and SW-846 8010/8020. She interprets chromatograms, calculates and reports results, and checks QC for these methods.

CYNTHIA WILSON

Chemist, Mobile Division

Ms. Wilson has a B.S. degree in chemistry from East Carolina University in Greenville, North Carolina. She joined Savannah Laboratories in 1989. She is responsible for the semivolatile organic analysis of environmental samples by GC/MS. Her duties include standard preparations, instrument calibrations, data interpretation and reporting, and maintenance of quality control for the GC/MS instrument.

Prior laboratory experience includes analytical testing to monitor plant operation for a municipal wastewater laboratory. She performed wet chemical analysis including BOD, DO, pH, specific conductivity, residual chlorine, chlorides, solids, and microbiological analysis for fecal and total coliforms. She previously worked at the Savannah, Georgia division of Savannah Laboratories as a GC volatiles chemist where she gained experience reporting volatile data and associated QC requirements.

Ms. Wilson has three years of analytical environmental testing experience on a variety of sample matrices including wastewaters, soils/sediments, and TCLP extracts. She is experienced in the use of purge and trap systems, FID, PID, and HECD detectors, and PE-Nelson and ChemStation data systems. She is familiar with SW-846 and 40 CFR Part 136 test methods, protocols, and QC requirements for semivolatiles organic analysis.

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PAUL CANEVARO

Laboratory Director/Project Manager, Deerfield Beach Division

Mr. Canevaro received his B.S. degree in chemistry from the University of Montevallo in 1982. He has a total of 12 years experience working in environmental laboratories. This experience includes the operation of GC, GC/MS, ICP, HPLC, and AA instruments using EPA protocols. Other related experience includes the coordination of inorganic and organic laboratories with a staff of 60 chemists and technicians, professional and technical guidance to laboratory supervisors, and development and maintenance of a quality control/quality assurance program.

Mr. Canevaro joined Savannah Laboratories in June, 1989 as the Deerfield Beach, Florida facility laboratory director. He is responsible for the day-to-day operation of the laboratory, project design and implementation, and client relations.

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RHONDA MOLL

Project Manager, Deerfield Beach Division

Ms. Moll received her B.S. degree in biology with a double minor in chemistry and physics from Troy State University. She began her career with Savannah Laboratories in 1987 at the Tallahassee Division where she determined volatile organic compounds using GC and GC/MS. Her past experience also includes the quality control testing and approval of raw materials used in the manufacture of pharmaceuticals and hospital supplies at Baxter Corporation in Miami, Florida.

Ms. Moll joined the Deerfield Beach Division upon its opening in 1989, as the volatile organics manager. She was responsible for the initial set-up of the volatiles laboratory which included instrumentation, quality control, methodology and personnel training. She coordinated all laboratory functions and reviewed all data generated from this department.

Ms. Moll was promoted to quality control manager of this division where she oversees all laboratory quality control functions such as evaluations, inspections, data review, and implementing new procedures required by methodology or Savannah Laboratories' corporate quality assurance program.

KATHY C. IRMINGER

Quality Assurance Manager, Deerfield Beach Division

Ms. Irminger received a B.A. degree in chemistry from Wake Forest University and began working at the Colorado School of Mines Research Center in Golden, Colorado. There she performed atomic absorption spectrophotometry in metallurgical and environmental applications and also developed inorganic bench methods. She also has worked for Camp, Dresser, and McKee and the Colorado State Department of Health performing inorganic EPA methods. At AC Laboratories in Florida, she developed expertise in gas chromatography for EPA Methods 601, 602, 502.2, 604, 610, 8010, and 8020.

Ms. Irminger joined Savannah Laboratories' Deerfield Beach Division when it opened in 1989, as a volatiles chemist performing GC and GC/MS analyses. She is well versed in SW-846 and CFR 40 methodologies.

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MARIANNE WALKER

Sample/Data Manager, Deerfield Beach Division

Ms. Walker's experience with environmental analytical laboratories includes two years at Pioneer Laboratory, Inc. in Pensacola, Florida, where she served as reporting department manager as well as office manager.

Ms. Walker joined Savannah Laboratories in 1989, where she assisted in coordinating the initial set-up of the Deerfield Beach laboratory operation. Her initial responsibilities included supervision of sample custody, sample bottle preparation, sample login, and data entry, as well as providing in-house project coordination.

As Sample/Data Manager, Ms. Walker is currently responsible for coordinating analytical programs for many of Savannah Laboratories' clients, including major consulting firms, counties, and water management districts.

JANICE WILTSHIRE

Data Coordinator, Deerfield Beach Division

Ms. Wiltshire obtained her diploma in computer science from Computer and Business Institute in Jamaica and has one year experience as a data entry clerk at AC Laboratories. She joined Savannah Laboratories in 1990, and her duties include all phases of data handling and assisting project mangers with client report preparation.

PHILL TAYLOR, JR.

Field Coordinator, Deerfield Beach Division

Mr. Taylor joined Savannah Laboratories in 1990. His previous experience includes working as a water treatment plant operator for the cities of Deerfield Beach and Pompano Beach, Florida. Mr. Taylor's current responsibilities include all field sampling activities scheduled at the Deerfield Beach facility.

KIMBERLY L. AMBISCO-KOSTZER

Organics Manager, Deerfield Beach Division

Ms. Ambisco-Kostzer has a B.S. degree in biology/pre-med with a minor in chemistry from Barry University of Miami Shores, Florida. She worked at the University of Miami School of Medicine as a research biochemist after graduation.

Ms. Ambisco-Kostzer joined Savannah Laboratories in 1989. Her duties include the analysis of pesticides, herbicides, phthalates, EDB, PAH, phenols, hydrocarbons, and formaldehydes.

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LINDA BACKUS

Chemist, Deerfield Beach Division

Ms. Backus received a B.S. degree in microbiology and a B.A. degree in chemistry from Florida Atlantic University. She taught microbiology at the collegiate level and performed extensive research on oral microbes. Upon graduation, she was employed with the University of Miami/Jackson Memorial Hospital initiating a tissue procurement facility for nationwide cancer research.

Ms. Backus joined Savannah Laboratories in 1990 as a trace metal analyst. Her present duties include determinations and data reporting of metals utilizing ICP techniques.

THERONA T. JAMES

Analyst, Deerfield Beach Division

Ms. James has a B.A. degree in chemistry with a minor in education from Columbia College in Columbia, South Carolina.

Prior to her employment with Savannah Laboratories in 1991, she was employed by the South Carolina Department of Environmental Health and Control as a chemist. There her duties encompassed wet chemistry, metals, and asbestos analyses. Her previous experience also includes metals analysis, digestion, and QA/QC data responsibilities at AC Laboratories in Fort Lauderdale, Florida.

Ms. James' current responsibilities at the Deerfield Beach facility include the analysis of volatile organic compounds by GC.

CATHERINE KATSIKIS

Analyst, Deerfield Beach Division

Ms. Katsikis received a B.S. degree in chemistry from the Chemical Engineering College in Athens, Greece. Her previous experience as a chemical laboratory supervisor included working with PVC stabilizers, polymerics, co-polymers, and organic compounds.

Ms. Katsikis joined Savannah Laboratories in 1990 as a trace metals analyst. Her duties include determinations and data reporting of metals utilizing ICP techniques.

ERIC SCHINSING

Technician, Deerfield Beach Division

Mr. Schinsing has been with Savannah Laboratories since 1990. He has an A.S. degree from the Community College of The Finger Lakes of New York and is currently pursuing a degree in oceanographic engineering. His responsibilities include assisting the analysts in the general chemistry laboratory.

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LAWRENCE TEICH

Chemist, Deerfield Beach Division

Mr. Teich has a B.A. in chemistry with an emphasis in zoology from Florida Atlantic University. Prior to joining Savannah Laboratories in 1989, he taught laboratory course work in animal physiology at Florida Atlantic University in Boca Raton.

Mr. Teich's responsibilities at Savannah Laboratories include mercury determinations by cold vapor atomic absorption. He also assists with trace metal analysis of As, Pb, Se, and Tl by graphite furnace atomic absorption and helps in the digestion of samples.

MARY VALEST

Chemist, Deerfield Beach Division

Ms. Valest obtained a B.S. degree in chemistry from the Catholic University of Puerto Rico. She completed her Chemistry Practicum at Destileria Serralles, Inc., a rum manufacturing company, where she performed analysis of finished products by UV, GC, and colorimeter. In raw materials, she conducted water monitoring, testing and distillation of fermented sugars.

Ms. Valest assisted on a temporary basis as quality control manager at Fruits Drinks, Inc. (Puerto Rico) where her responsibilities included the quality control process, documentation and daily production reports; coordination of chemical and microbiological testing of raw materials and finished products; supervision of laboratory technicians; and control of safety and sanitation systems.

She then joined SmithKline Beecham Pharmaceuticals (Puerto Rico) where she worked as quality assurance analyst with bulk, finished products, and long term stability samples conducting testing procedures by HPLC, GC, UV, turbidimeter, osmometer and extractions. In the area of raw materials, she worked with IR, TLC, gravimetric analysis, all process sterile water monitoring and process rinse water testings.

In 1990, she moved into the continental United States and worked on a temporary basis with Schering-Plough (Pembroke Pines, Florida) where she performed dissolution technology and HPLC and assisted in identification of chromatographic samples and calculation of resolution and tailing factors for a new quantitation method for fatty acids.

Ms. Valest joined Savannah Laboratories in 1991, and she is currently working in the semivolatile GC department performing EDB and pesticide residue analysis.

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CAROL-ANN VASSELL

Chemist, Deerfield Beach Division

Ms. Vassell obtained a B.S. degree in chemistry from the City College of New York. Upon graduation, she worked as a research chemist at the Institute of Food and Agricultural Sciences.

- Ms. Vassell joined AC Laboratories where she performed trace metal determinations by AA as well as EDB and trihalomethane determinations by GC. She also conducted sample extractions for organic compounds as well as chloride, cyanide, and phenols determinations.
- Ms. Vassell joined Savannah Laboratories in 1990 as a chemist. Her responsibilities include the analysis of acids, base neutral and pesticide compounds by GC/MS techniques.

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KATHY SHEFFIELD

Laboratory Director/Project Manager, Tampa Bay Division

Ms. Sheffield has B.S. degrees in chemistry and biology plus seven years of analytical chemistry experience. Throughout her career, she has specialized in the field of organic chemistry and has developed considerable expertise in pesticide and volatile organic analysis.

Ms. Sheffield was employed by the Florida Department of Agriculture in the Pesticide Use Monitoring Section of the Chemical Residue Laboratory. She was responsible for the analysis of water, soil, and food products for a variety of pesticides and herbicides using gas chromatography and high performance liquid chromatography (HPLC). She was instrumental in the development of new procedures using HPLC, and wrote several standard operating procedures. Ms. Sheffield was promoted to a supervisory level where she was responsible for all data generated by the analytical section.

Ms. Sheffield began her employment with Savannah Laboratories and Environmental Services, Inc. as senior chemist. She performed analyses on environmental samples using instrumental techniques including GC/MS, GC, and HPLC. She was promoted to organic section manager, with additional duties in the administration of the quality assurance program.

As laboratory director/project manager, Ms. Sheffield is responsible for coordinating analytical programs for many of Savannah Laboratories' clients, including major consulting firms, counties, governmental agencies, and industries.

ANDRE RACHMANINOFF

Project Manger, Tampa Bay Division

Mr. Rachmaninoff has a B.A. degree in biology from Kalamazoo College and eight years of experience in environmental analytical work. Six years of this experience was in a supervisory or laboratory management capacity.

Mr. Rachmaninoff is thoroughly familiar with analytical, microbiological, and radiochemistry techniques. He has extensive bench experience in both flame and furnace AA spectroscopy, ICP emission spectroscopy, IR and UV/VIS spectroscopy, Gamma spectrometry, alpha and beta particle emissions analysis, and numerous automated and manual wet chemistry analyses.

Mr. Rachmaninoff is an active member of the Florida Society of Environmental Analysts, and served as President from 1990-1991.

Mr. Rachmaninoff provides technical assistance and support to clients for field and analytical services as a project manager.

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DOMINIC P. FRALLI

Project Manager, Tampa Bay Division

Mr. Fralli holds an M.S. degree in environmental science from the University of Texas in Dallas. He has more than seven years experience as a gas chromatography chemist and supervisor. Mr. Fralli was an environmental scientist with the Hillsborough County Environmental Protection Commission where he was responsible for the operation of the organic section of the laboratory prior to joining Savannah Laboratories. He implemented EPA methods for analysis of water and soil samples and was in charge of the QA/QC generated for these methods. He supervised two chemists and provided information concerning organic chemicals to the public and to agency personnel.

As QA Manager, he is responsible for ensuring that method QA requirements are met and also issues and evaluates in-house check samples.

TRACY H. BOTTO

Inorganics Manager, Tampa Bay Division

Ms. Botto holds a B.S. degree in microbiology from the University of Maine and has five years experience in a variety of laboratory techniques. Her specialty is trace metals determinations by ICP, though she is familiar with flame, furnace, and cold-vapor techniques.

Her responsibilities as inorganics manager include management of personnel, overseeing of all method development, and adherence to EPA methodology and QA/QC requirements of the Tampa Bay Division laboratory.

INAS M. SOBKY

Quality Assurance Manager, Tampa Bay Division

Ms. Sobky has a B.S. degree in chemistry/zoology from Ain Shames University, Cairo, Egypt and seven years of experience in organic environmental analytical work. Four years was spent in an organic laboratory management capacity.

Ms. Sobky is familiar with EPA 500, 600, and 8000 series gas chromatography (GC) methodologies. She has additional training from ACS in gas chromatography system maintenance and troubleshooting. She attended an analytical gas chromatography workshop with the Southeastern Chromatography Association and she is an active member of the Florida Society of Environmental Analysts.

Ms. Sobky's responsibilities include management and implementation of technical EPA GC methods, training and supervision of organic chemists and technicians, and maintenance of GC instrumentation as a GC manager for Savannah Laboratories, Tampa Bay Division.

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LINDA DOWD

Analyst, Tampa Bay Division

Ms. Dowd has a B.S. degree in biology from York College and an A.S. degree in environmental health technology from Queensboro Community College. While working toward her degrees, she was employed as an environmental educator for the New York City Department of Parks. Her duties included flora and fauna identification and inventories, water sampling, and the creation and implementation of a variety of environmental education programs for the public.

Her responsibilities as an analyst include the preparation of standards, calibration and loading of the GC, and assisting the chemist in charge with identification and calculation of volatile organic compounds for the volatiles section at Savannah Laboratories.

CHRIS E. HARRIS

Field Sampler/Lab Technician, Tampa Bay Division

Mr. Harris has more than three years of experience as a field/laboratory technician in the environmental field. He has collected soil, groundwater, surface water, drinking water, and industrial waste samples using current EPA protocol. In the laboratory, he has performed a wide variety of titrimetric, gravimetric, and colorimetric analyses according to EPA protocol for both aqueous and nonaqueous sample matrices.

Mr. Harris is responsible for the scheduling and planning of all field sampling projects for the Tampa Bay facility. When not collecting field samples, he is responsible for all analyses associated with the general and biological laboratories.

CARL JOHN HOOVER, JR.

Chemist, Tampa Bay Division

Mr. Hoover holds a B.S. in zoology from the University of South Florida. He has more than six years experience in the environmental field. Mr. Hoover was a chemist with PBS&J Environmental Laboratories where he was responsible for trace metals analysis by GFAA, hexavalent chromium analysis, mercury analysis by cold vapor method and TCLP extractions prior to joining Savannah Laboratories. He was an environmental scientist and laboratory technician at Southwest Florida Water Management District where he assisted in environmental impact studies and performed wet chemistry analysis before his employment with PBS&J.

At Savannah Laboratories, Mr. Hoover is responsible for trace metals analysis by GFAA and by ICP.

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ANTONIUS LEBRUN

Chemist, Tampa Bay Division

Mr. Lebrun has a B.S. degree in chemistry from University of Florida. He was a chemist with Cargill/Gardinier, Inc., in Riverview, Florida, where he worked in the environmental department prior to joining Savannah Laboratories.

At Savannah Laboratories, Mr. Lebrun is responsible for analysis of samples for metals by ICP and GFAA.

MARSHA MARTINOVICH

Analyst, Tampa Bay Division

Ms. Martinovich has a B.A. degree in sociology with a minor in chemistry from West Virginia University in Morgantown, West Virginia, and has more than three years experience in the environmental field. She was responsible for analysis of organic contaminants in water, wastewater, soil, and hazardous waste samples prior to joining Savannah Laboratories as a GC analyst.

At Savannah Laboratories, Ms. Martinovich is responsible for volatile analysis by GC using methods 601, 602, 8010, and 8020.

NATALIE L. PARK

Analyst, Tampa Bay Division

Ms. Park has a B.S. degree in biology from Florida State University. She has two years experience doing organic extractions at an environmental laboratory. She was responsible for extraction of soil and water samples for EPA Methods 604, 606, 608, 610, 614, 615, 625, and all SW-846 series methods. She is also familiar with Methods 418.1 and 413.2.

Ms. Park's duties at Savannah Laboratories include metal digestions, TCLP extractions, and mercury analysis using cold vapor techniques.

General Engineering Laboratories, Inc.

Quality Assurance Plan

This document conforms to the guidelines established by Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, EPA QAMS 005/80.

DOCUMENT GL-QS-B-001 REVISION 9

April 8, 1994

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Effective Date: April 8, 1994
Signature Page

APPROVAL SIGNATURES

General Engineering Laboratories, Inc. (GEL), Quality Assurance Plan has been prepared in accordance with the Environmental Protection Agency's "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80. This plan is part of GEL's Corporate Quality System and is specific to sample collection and laboratory practices. This plan is applicable to GEL laboratory operations as well as those of Environmental Physics, Inc. (EPI), a GEL affiliate, which operates under the same quality program.

Approved by:

Robert L. Pullano

Manager, Quality Systems

General Engineering Laboratories, Inc.

James T. Chambers

Manager, Laboratory Services,

General Engineering Laboratories, Inc.

James B. Westmoreland

Manager, Radiochemical Services,

Environmental Physics, Inc.

Date

Date

8 April

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I. INTRODUCTION TO ANALYTICAL SERVICES

Α. Company Profile

General Engineering Laboratories, Inc. (GEL) is an environmental services company incorporated in South Carolina in 1981 to provide analytical and environmental consulting services to industrial and municipal clients, government agencies, and other environmental consultants and analytical laboratories nationwide. GEL offers a comprehensive range of analytical, environmental consulting, and field services. Using state-of-the-art equipment and methods, the analytical division provides a comprehensive array of organic and inorganic analyses and related support services to meet our clients needs. Located in the same facility, Environmental Physics, Inc. (EPI), a GEL affiliate, provides laboratory services for multi-media, radiochemical analyses in support of the nuclear and non-nuclear industry.

This Quality Assurance Plan has been prepared in accordance with the Environmental Protection Agency's QAMS-005/80 "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans". This plan is part of GEL's Corporate Quality System and is specific to sample collection and laboratory practices. The Corporate Quality System is designed to conform to requirements outlined in the following standards:

- ASME/ANSI NQA-1
- ISO 9001/O91
- ISO/IEC GUIDE 25

The company's corporate quality policies are detailed in the corporate Quality Manual GL-QS-A-001.

В. Description of Facilities

The GEL and EPI laboratories are structured to provide dedicated space for radiochemistry analyses, organic extractions, semi-volatile organics analyses, volatile organics analyses, metals analyses, general chemistry analyses, and air analyses. The laboratories and offices occupy 73,000 square feet specifically designed to meet the stringent quality control and utility requirements of the modern environmental laboratory. A diagram of the present building layout is presented in Appendix 1.

The air conditioning system controls the environment in individual laboratories to ensure optimum performance of sensitive instruments and to eliminate the potential of cross contamination. Negative and positive pressure air locks isolate the individual laboratories to ensure that airborne contaminants do not enter these laboratories. The partition walls in the laboratory are constructed of wire-reinforced glass to provide high visibility and a safe working environment for the analysts. An extensive list of utilities is provided at each work station in the laboratory to ensure that analytical procedures are carried out efficiently and in the most cost effective manner.

The conditions under which the sample is stored and the manner in which it is handled by the laboratory are important considerations. The entire laboratory is a secured area. A coded identity card is required to gain access to the laboratory and a computer

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record is maintained of people entering the laboratory. Visitors must be escorted through the laboratory by authorized company personnel.

When analyses are not being conducted, the samples are stored in specially designated storage rooms. Samples subject to degradation at ambient temperatures are stored in a specially designed refrigerated room. Samples not requiring refrigeration, such as oils and hazardous waste materials, are stored in a separate room kept at ambient temperature. EPA sample storage requirements are found in Appendix 2.

With the exception of the cold room used for radioactive samples, all sample storage rooms are located within the secured laboratory area. The radioactive sample cold room is located immediately behind the south wing of the building. The door to this room is locked and the key is kept in the possession of the log-in personnel. Access to the storage rooms is restricted to authorized personnel. Samples are stored for at least 45 days after completion of the analysis and until the sample is ready for proper disposal.

Safety is an important feature of the laboratory. The facility is equipped with a fire alarm system designed to detect smoke in certain high risk areas. These areas are equipped with a bell and buzzer warning system and an automatic halon extinguisher. The areas covered by the halon system are the cold storage room, ambient storage, organic preparation laboratory, hazardous waste laboratory, and flammable storage. In addition, dry chemical fire extinguishers are located at strategic points throughout the building. Special cabinets have been installed for the storage of acids and solvents that are used on a daily basis. A concrete flammable storage room located next to the stock room is used for bulk storage of solvents.

Safety equipment is available to all employees as needed including safety glasses, lab coats, safety goggles, protective gloves, hard hats, and coveralls. Eye wash stations and overhead showers are located throughout the laboratory in the event of accidents.

While laboratory personnel encounter insignificant levels of occupational radiation exposure, Thermo Luminescent Dosimeters (TLD) are placed in all laboratory areas and on selected laboratory and field personnel to monitor radiation exposure for added assurance. All TLDs are exchanged on a quarterly basis and analyzed by a NAVLAP approved laboratory for exposure measurement. Exposure records are maintained by the Radiation Protection Officer.

С. Utility Services

The following utilities are provided in each laboratory:

- Cold Water
- Hot Water
- Deionized Water
- Compressed Air
- Natural Gas
- Vacuum
- 110 Volt AC
- 220 Volt AC (at selected stations)

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Deionized Water

Deionized water is distributed throughout the laboratory through half inch schedule 40 PVC pipe. This water is made from city water by a Continental Services Deionization System capable of producing 20 gallons per minute (gpm) of Type II laboratory water.

At a minimum, the laboratory water is analyzed monthly for the following parameter specifications:

•	Conductivity	<2.0 μmnos/cm @ 25° C
•	pH	5.5 - 7.5
•	ŤOC	<1.0 mg/L
•	Trace Metals, Single (Cd, Cr,	_
	Cu, Ni, Pb, Zn)	<0.05 mg/L
•	Trace Metals, Total	<0.1 mg/L
•	Free Chlorine	<0.1 mg/L
•	Standard Plate Count	<1000 cfu/ml
•	Ammonia/organic nitrogen	<0.1 mg/L

D. Equipment

Various support equipment is available in the laboratory. Proper operation of the laboratory equipment is essential for efficient and reliable operation of the laboratory. Routine maintenance and care of the equipment minimizes down time and assists in providing quality data.

The following is a general list of the support equipment found in GEL's various laboratories.

- Autoclaves
- Automatic Pipettes
- Thermometers
- Ultraviolet Sterilizer
- Water Baths
- Grinders and Homogenizers
- Hot Plates and Heating Mantles
- Incubators
- Microscopes
- Muffle Furnaces
- Ovens
- Distillation Apparatus
- Filtration Apparatus
- Selective Ion and pH Meters

Balances

All balances used at GEL are on a service contract for annual calibration, maintenance, and cleaning. Each balance is labeled with the balance serial number, the service date, the date of the next scheduled service, and the signature of the service technician. GEL maintains a set of class S weights that are traceable to NIST. These weights are calibrated annually by the South Carolina Department of Agriculture.



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Analysts follow GEL Standard Operating Procedure (SOP) GL-LB-E-002 for Balances for the set-up, calibration, and operation of balances. The procedure identifies the use of proper balance set-up, based upon the requirements set by the analytical method being used. The SOP describes the daily calibration verifications using weights verified against class S or higher weights. It also states the requirements for the routine operation for the balances, and troubleshooting when calibration verifications fall outside of the control limits specified in each balance log.

Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices

Laboratory refrigerators and freezers are used to store samples, standards, and other temperature sensitive materials. Federal regulations and client specifications require that certain samples be maintained within sub-ambient temperature ranges. Sample temperature control is essential to the production of legally defensible data. Refrigerators and freezers must be monitored to ensure that temperatures are within acceptance criteria. Documentation is required through the completion of monitoring logs to assure that control was sufficient. Monitoring logs are posted on the refrigerators and freezers.

Careful temperature control is central to the production of acceptable data. Temperature excursions beyond established limits may invalidate the procedure and associated data. Incubators, ovens, waterbaths, and similar devices used during the temperature sensitive portion of those analytical procedures must be monitored to ensure compliance with analytical requirements. Temperature control during process completion must be documented on monitoring logs.

The requirements for monitoring and recording temperatures of refrigerators, freezers, ovens, incubators, waterbaths, and similar devices are explained in GEL SOP GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices. Instructions are provided for general requirements of temperature monitoring devices; required documentation; the responsibilities of the analysts, Group Leaders, Process Control Engineer, and Laboratory Manager, and the action required for out of control conditions.

E. INSTRUMENTATION

Accurate and reliable analysis in the laboratory is ensured by the use of Environmental Protection Agency (EPA) and Department of Energy (DOE) approved analytical procedures and advanced instrumentation. Training sessions are held to familiarize the analysis with new methodologies. Proper use and care of the instrumentation in the analytical laboratory is essential for the development of accurate and reliable data.

All instrumentation is connected to an Uninterrupted Power System (UPS). The following is a list of GEL instrumentation. It is organized by analysis type and includes the purchase date and serial number for each instrument.

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INSTRUMENTATION	DATE	SERIAL #
Organic Extractions Organomation Continuous Extractors, modified	6/7/90 6/7/90 6/7/90	1024 1025 1026
Semi-Volatile Organics Tracor 540 Gas Chromatograph with Autosampler and Dual Electron Capture Detectors	6/1/87 2/7/90 5/14/91	871138 891681 911864
Tracor 560 Gas Chromatograph with Autosampler and Dual Electron Capture Detectors	1983	2083-81
Hewlett-Packard 5970 Gas Chromatograph/Mass Spectrometer with Autosampler	7/10/89 7/10/89 1/15/90 10/3/91	2905A11951 2905A11952 2923A12409 3114A13052
Hewlett-Packard 5972 Gas Chromatograph/Mass Spectrometer with Netech Air Cryogenic Concentrator	1/94 4/93	3251A00153 3341A00979
Perkin Elmer HP Liquid Chromatograph Tracor 540 Gas Chromatograph with Autosampler	10/8/89 8/15/88	20649
and Dual Flame Ionization Detectors		881397
Tracor 560 Gas Chromatograph with a Flame Ionization Detector	8/1/87	1857-80
Hewlett-Packard 5890 Gas Chromatograph with Autosampler and Dual Flame Ionization Detectors	3/92	3203A41418
Volatile Organics	10/01/07	0.41074
Tracor 540 Gas Chromatograph with Purge & Trap Autosampler, Hall Electron Capture Detector, and	12/31/86 9/1/85	861076 851320
Photoionization Detector Hewlett Packard 5972 Gas Chromatograph/Mass	1/19/90 4/93	891691 3341A00976
Spectrometer with Purge & Trap Autosampler Hewlett-Packard 5970 Gas Chromatograph/Mass	1/94 11/15/90	3251A00145 3004A12641
Spectrometer with Purge & Trap Autosampler	7/1/91	3034A12910
	<i>7/1/</i> 91	3034A12909
Metals Thermo Jarrell Ash ICP 61 E with Autosampler	11/15/90	78090
Thermo Jarrell Ash Enviro-II Simultaneous/Sequential ICP with Autosampler	7/93	170390
Perkin Elmer 5100 Graphite Furnace Atomic	12/27/88	134724
Absorption Spectrophotometer with Autosampler Thermo Jarrell Ash SH 4000 Graphite Furnace Atomic Absorption Spectrophotometer with Autosampler	7/15/89 11/15/91	136462 8156
Buck 400 Cold Vapor Mercury Analyzer	2/8/89	45
Bacharach MAS-50 Cold Vapor Mercury Analyzer	5/10/90	UB0520

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INSTRUMENTATION	DATE	SERIAL #
Leeman PS 200 Cold Vapor Mercury Automatic Analyzer	9/22/93	HG3063
General Chemistry		
Dohrman DC 80 Total Organic Carbon Analyzer with Autosampler	11/30/82	9604
Dohrman DC 190 Total Organic Carbon Analyzer with Autosampler	5/93	9302155
Mitsubishi TOX-10-C Total Organic Halogen	7/16/84	43R00334
Analyzer Dohrman DX 2000 Total Organic Halogen Analyzer	1/23/90 10/93	43R31429 9309876
with Autosampler		
Technicon Traacs 800 Automated Chemistry Autoanalyzer	3 <i>/</i> 7/90	165-A011-02
Alpkem 510 Automated Chemistry Autoanalyzer	1984	141
Dionex 4500i Ion Chromatograph	6/1/89	891401
Dionex DX300 Ion Chromatograph	3/93	930419
Perkin Elmer 1600 Fourier Transform Infrared	11/88	134908
Spectrophotometer EM Science Karl Fischer Moisture Analyzer	1986	85105-09
Alpkem 510 Automated Chemistry Flow Solution	9/14/90	540
System	3.1-1.30	5-0
Radiochemistry		
Tennelec LB-5100 Proportional Counters	1/91	62804
	8/92	14740
Tennelec LB-4100 Proportional Counter with 24	3/93	18483
Detectors	6/93	21938
Canberra HT-1000-D Alpha/Beta Counter and 8	11/91	10912771
Detectors Beckman LS 6500 Series Liquid Scintillation	6/93	7065155
Counters	6/93	7067083
	4/94	7067404
Canberra GC 3519 High Efficiency Germanium	12/91	5933088
Detector for Gamma Spectroscopy	12/91	11912863
• • •	1/93	12922955
	6/93	5933105
	1/94	1943199
	1/94	1943234
Canberra GX 3519 Extended Range High Efficiency	8/93	8932581
Germanium Detector for Gamma Spectroscopy	11/93	10935015
Canberra GCW 3522 Germanium Well Detector for	4/94	3941466
Gamma Spectroscopy Canberra GL 2020/S Low Energy Germanium	1/93	5933105
Detector for X-Ray Spectroscopy		
Canberra Alpha/Gamma Data Management System	2/92	G-4770

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INSTRUMENTATION	DATE	SERIAL #
Canberra PA45020 Alpha PIPS Detectors for Alpha	2/92	13258
Spectroscopy	2/92	13259
operation (2/92	13260
	2/92	13911
	2/92	19312
•	2/92	13261
	2/92	17940
·	2/92	13266
	2/92	13268
	2/92	13269
	2/92	13270
	2/92	13271
	2/92	13272
	2/92	13273
	2/92	13274
	2/92	13275
	2/92	13296
·	2/92	13912
	6/93	17024
	6/93	16319
	6/93	16320
	6/93	19313
	6/93	16323
	6/93	16326
	10/93	18915
	10/93	18916
	10/93	18917
	10/93	18918
	10/93	18937
	10/93	18938
	10/93	18939
	10/93	18941
	10/93	18942
	10/93	18943
	10/93	19310
	10/93	19311
Ludlum Model 200 Scalers for Radium 226 Analysis	5/92	101846
	5/92	86493
	6/93	86494
	10/93	104617
Support Systems		
Perkin Elmer Nelson 3000 Series Data Acquisition	Nov-90	333572879
System	Nov-90	333572885
-	Nov-90	333572881
	Nov-90	112573508
	Nov-90	333572882
	Nov-90	333572883
	Nov-90	333572884
	Nov-90	333572880
	-	

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INSTRUMENTATION	DATE	SERIAL #	
	Nov-90	333572878	
	Nov-90	333582875	
Laboratory Information Management System	Dec-90	051K0015	

Laboratory Information Management Systems (LIMS)

Computers and peripheral equipment (terminals, plotters, printers, and tape drives) are used to monitor the status of laboratory tests, control instruments, evaluate accuracy and precision of laboratory results and prepare client reports. This is managed by the Laboratory Information Management System (LIMS) which is integral to the daily operation of the laboratory.

GEL's advanced, networked LIMS is uniquely flexible. GEL's system utilizes state-of-the-art hardware, software, and networking to maximize the ability to quickly access client information. Sample tracking, data acquisition, quality control review, and generation of data reports are performed on a dual Sun SparcServer® 4/470 system utilizing a UnixTM based operating system. Apple MacintoshTM computers linked to the LIMS are used for statistical evaluations, engineering design work, project scheduling, word processing, and custom report formatting.

Redundant primary information systems ensure maximum LIMS system availability. Pairs of all major computer and data storage units are in place and mirroring software duplicates all data by storing it to multiple locations simultaneously.

Samples received by the laboratory are entered immediately into LIMS. The LIMS software assigns a unique laboratory sample identification number, as well as a code for each requested analysis, to individual samples. Worksheets are then generated by the software and are automatically prioritized to alert the analyst of work which must be completed by a particular deadline. Once the requested analyses have been performed, analytical results are entered directly into the computer database either by automatic data transfer or the analyst.

After the results are reviewed and released by the Quality Department, the analytical report is printed onto a Certificate of Analysis form. Although data is routinely printed in a standard certificate format, the LIMS system allows custom reports to be generated in order to meet individual clients' specific needs.

Apple MacintoshTM computers are linked to the LIMS via an ethernet gateway and perform all terminal functions. They are also used in statistical evaluations, engineering design work, project scheduling, word processing, and custom report formatting. A computer security system prevents inadvertent or unauthorized access to the various elements of the laboratory database.

F. Glassware

Glassware-cleaning procedures vary by the type of analysis. These procedures are described in GEL SOP GL-LB-E-003 Glassware Preparation. The SOP describes the

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proper washing, soaking, rinsing, and storage techniques for reusable bottles and plasticware. It also describes cleaning techniques for glassware used for the analysis of Mercury, Biochemical Oxygen Demand, Organics, Metals, General Chemistry, MBAS, Total Organic Halogens, Phosphate, and TKN.

G. Analytical Standards

Analytical Reagent Grade (AR) chemicals are used for most analyses in the inorganic laboratory. Ultrapure or pesticide grade solvents are used for organic analyses. If the purity of a reagent is not specified by the procedure, AR Grade chemicals are used.

Standards are prepared according to GEL SOP GL-OA-E-002 for Organic Standards Preparation and Traceability, GL-MA-E-004 for Metals Standards, and GL-GC-E-004 for Standards - Definitions and Preparation. Radioactive standards are prepared according to GL-EPI-E-M001. Information concerning the preparation of standards is recorded in standard logbooks following GEL SOP GL-LB-E-007 for Laboratory Standards Documentation.

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II. LABORATORY ORGANIZATION AND RESPONSIBILITY

The company organizational charts for GEL are shown in figures 1, 2 and 3. These charts represents the flow diagram of the chain of command and responsibility for all sections of the company.

Molly F. Greene and George C. Greene III, P.E., Ph.D., are joint owners of General Engineering Laboratories, Inc. Together they share the ultimate responsibility for all facets of company operations. The individual service groups, quality, and the advisors report directly to the Greenes' through the responsible manager as shown in the company organizational chart.

Resumes for key professionals in the organization are included in Section XV. These resumes provide summaries of the specific duties performed by each individual, professional qualifications, including formal education as well as special training, and experience related to the employee's specific function in the company.

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GENERAL ENGINEERING LABORATORIES, INC.

MANAGEMENT ORGANIZATION

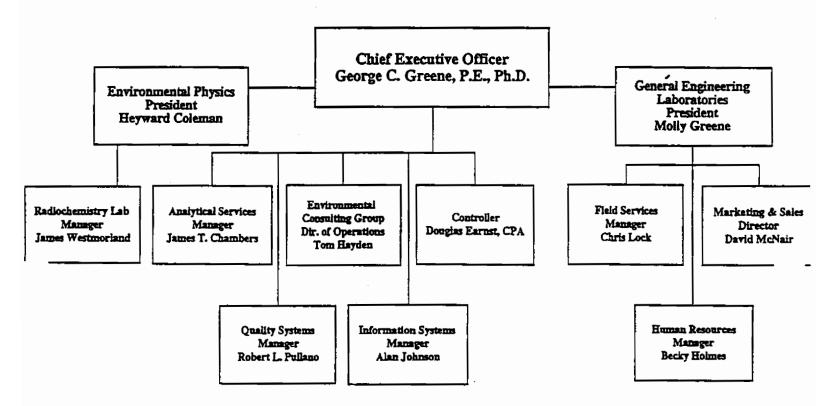


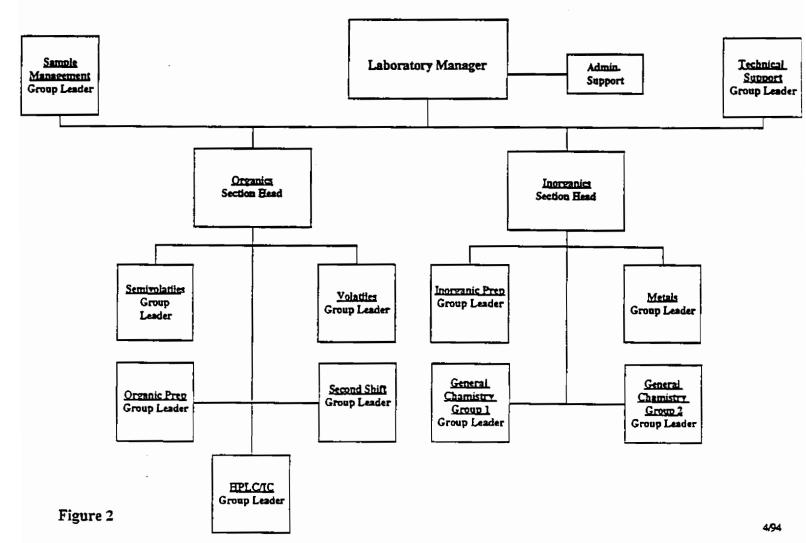
Figure 1

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GENERAL ENGINEERING LABORATORIES, INC.

ANALYTICAL SERVICES



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ENVIRONMENTAL PHYSICS, INC. RADIOCHEMICAL SERVICES

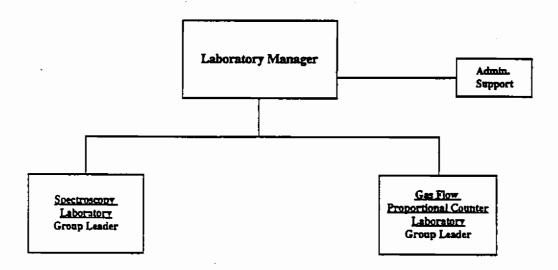


Figure 3

4/94

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III. QA/QC OBJECTIVES

The parameters analyzed by GEL are divided into four main categories:

- Organic Analyses
- Metals Analyses
- General Inorganic Analyses
- Radiochemistry Analyses

For each parameter in these categories, it is essential to establish limits of acceptance for Quality Control (QC) data. QA/QC objectives for precision, accuracy, and completeness have been established for specific analytes in each method. The definitions of precision, accuracy, and completeness are as follows:

- Precision A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".
- Accuracy Accuracy is a measure of the bias of a system. The degree of
 agreement of a measurement (or an average of measurement of the same thing),
 X, with an accepted reference or true value, T, usually expressed as the
 difference between the two values, X-T, or the difference as a percentage of
 the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio,
 X/T.
- Completeness A measure of the amount of data obtained from a measurement process compared to the amount that was expected to be obtained under the conditions of measurement.

All measurements must be made such that results are representative of the media (i.e. surface water, groundwater, soil/sediment, solid waste, etc.) and conditions being measured.

The accuracy and precision control limits for each of the parameters in the four categories for analysis are based on:

- Historical Data
- Replicate Analyses
- Spike Analyses
- Standard Analyses

General Engineering Laboratories, Inc. Quality Assurance Plan

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The Data Quality Objectives are documented in Tables 1 through 5 of this section for the following parameters:

DW = Drinking Water SW = Surface Water GW = Groundwater

SED = Sediment

S = Soil EFF = Effluent

HW = Hazardous Waste (including chemical wastes and sludges)

SA = Saline Water V = Vegetation AF = Air Filter W = Water

Table 1 Data Quality Objectives - Organics

Method No	. Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
EPA 8240	GW	Acetone	NA	NA	
	• • • • • • • • • • • • • • • • • • • •	Acrolein	NA	NA	
		Acrylonitrile	NA	NA	
		Benzene	S11	76-127	95
		Bromodichloromethane	NA	NA	93
		Bromoform	NA NA	NA NA	
		Bromomethane	NA NA	NA NA	
		Carbon Disulfide	NA	NA NA	
		Carbon Tetrachloride	NA NA		
		Chlorobenzene		NA 75 120	05
		Chloroethane	≤13	75-130	95
			NA	NA	
		2-Chloroethylvinyl ether	NA	NA	
		Chloroform	NA	NA	 .
		Chloromethane	NA	NA	
		Dibromochloromethane	NA	NA	
		1,2-Dichlorobenzene	NA	NA	
		1,3-Dichlorobenzene	NA	NA	
		1,4-Dichlorobenzene	NA	NA	
		Dichlorodifluoromethane	NA	NA	
		1,1-Dichloroethane	≤14	61-145	95
		1,2-Dichloroethane	NA	NA	
		1,1-Dichloroethene	NA	NA	
		1,2-Dichloroethene	NA	NA	
		1,2-Dichloropropane	NA	NA	
		c-1,3-Dichloropropene	NA	NA	
		t-1,3-Dichloropropene	NA	NA	
		Ethylbenzene	NA	NA	
		2-Hexanone	NA	NA	
		Methylene Chloride	NA	NA	
		Methyl Ethyl Ketone (MEK)	NA	NA	
		4-Methyl-2-pentanone (MIBK)	NA ·	NA	
		1,1,2,2-Tetrachloroethane	NA	NA	
		Tetrachloroethene	NA	NA	
		Toluene	≤13	76-125	95
		1,1,1-Trichloroethane	NA	NA	
		1,1,2-Trichloroethane	NA NA	NA	
		Trichloroethene	≤ 21	62-137	95
		Trichlorofluoromethane	NA	NA	93
		Styrene	NA	NA NA	
		Vinyl Acetate	NA NA		
		Vinyl Chloride		NA	***
		o-Xylene	NA NA	NA	
		m,p-Xylenes	NA NA	NA	
		m,p-2xytomos	NA	NA	
EPA 8240	SED	Acetone	374	37.4	
LI /\ 024U	SED	Accetone	NA	NA	
	S	Acrolein	NA	NA	

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Table 1
Data Quality Objectives - Organics

Method No	. Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness %
	HW	Acrylonitrile	NA	NA	
	****	Benzene	NA ≤2 1	66-142	95
		Bromodichloromethane	NA	NA	93
		Bromoform	NA NA	NA NA	
		Bromomethane	NA NA		_
		Carbon Disulfide		NA	_
		Carbon Tetrachloride	NA NA	NA	
		Chlorobenzene	NA	NA	0.5
			≤21	60-133	95
		Chloroethane	NA	NA	
		2-Chloroethylvinyl ether	NA	NA	
		Chloroform	NA	NA	****
		Chloromethane	NA	NA	
		Dibromochloromethane	NA	NA	
		1,2-Dichlorobenzene	NA	NA	
		1,3-Dichlorobenzene	NA	NA	-
		1,4-Dichlorobenzene	NA	NA	
		Dichlorodifluoromethane	NA	NA	
		1,1-Dichloroethane	≤22	59-172	95
		1,2-Dichloroethane	NA	NA	
		1,1-Dichloroethene	NA	NA	
		1,2-Dichloroethene	NA	NA	
		1,2-Dichloropropane	NA	NA	
		c-1,3-Dichloropropene	NA	NA	
		t-1,3-Dichloropropene	NA	NA	
		Ethylbenzene	NA	NA	
		2-Hexanone	NA	NA	
		Methylene Chloride	NA	NA	****
		Methyl Ethyl Ketone (MEK)	NA	NA	
		4-Methyl-2-pentanone (MIBK)	NA	NA	
		1,1,2,2-Tetrachloroethane	NA	NA	
		Tetrachloroethene	NA	NA	
		Toluene	≤2 1	59-139	95
		1,1,1-Trichloroethane	NA	NA	
		1,1,2-Trichloroethane	NA	NA	
		Trichloroethene	≤14	71-120	95
		Trichlorofluoromethane	NA	NA	
		Styrene	NA	NA	
		Vinyl Acetate	NA	NA	
		Vinyl Chloride	NA	NA	
		o-Xylene	NA	NA	
		m,p-Xylenes	NA	NA	
EPA 624	sw	Benzene ·	NA	37-151	95
	GW	Bromodichloromethane	NA	35-155	95
	EFF	Bromoform	NA NA	45-169	95 95
	T-Y-T.	Bromomethane	NA NA	D-242	95 95
		Diomonicalanc	1477	17-444),)

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Table 1
Data Quality Objectives - Organics

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy %R	Completeness <u>%</u>
	Carbon Tetrachloride	NA	70-140	95
	Chlorobenzene	NA	37-160	95 95
	Chloroethane	NA	14-230	95 95
	2-Chloroethylvinyl ether	NA NA	D-305	95 95
	Chloroform	NA NA	51-138	95 95
	Chloromethane	NA NA	D-273	95 95
-	Dibromochloromethane	NA	53-149	95 95
	1,2-Dichlorobenzene	NA	18-190	95 95
	1,3-Dichlorobenzene	NA	59-156	95 95
	1,4-Dichlorobenzene	NA	18-190	
	1,1-Dichloroethane	NA	59-155	
	1,2-Dichloroethane	NA.	49-155	
	1,1-Dichloroethene	NA NA	D-234	95 95
	t-1,2-Dichloroethene	NA NA	54-156	
	1,2-Dichloropropane	NA	D-210	95 05
	c-1,3-Dichloropropene	NA NA		95 05
	t-1,3-Dichloropropene	NA NA	D-227	95 05
	Ethylbenzene		17-183	95 95
	Methylene Chloride	NA	37-162	95 95
	1,1,2,2-Tetrachloroethane	NA	D-221	95 2
	Tetrachloroethene	NA	46-157	95
	Toluene	NA	64-148	95
		NA	47-150	95 25
	1,1,1-Trichloroethane	NA	52-162	95
	1,1,2-Trichloroethane Trichloroethene	NA	52-150	95
	Trichlorofluoromethane	NA	71-157	95 05
		NA NA	17-181	95 05
	Vinyl Chloride	NA	D-251	95
EPA 524.2 DW	Benzene	<20	80-120	95
	Bromobenzene	<20	80-120	95
	Bromochloromethane	<20	80-120	95
	Bromodichioromethane	<20	80-120	95
	Bromoform	<20	80-120	95
	Bromomethane	<20	80-120	95
	n-Butylbenzene	<20	80-120	95
	sec-Butylbenzene	<20	80-120	95
	tert-Butylbenzene	<20	80-120	95
	Carbon Tetrachloride	<20	80-120	95
	Chiorobenzene	<20	80-120	95
	Chloroethane	<20	80-120	95
	Chloroform	<20	80-120	95
	Chloromethane	<20	80-120	95
	o-Chlorotoluene	<20	80-120	95
	p-Chlorotoluene	<20	80-120	95
	Dibromochloromethane	<20	80-120	95
	1,2-Dibromoethane	<20	80-120	95

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Table 1
Data Quality Objectives - Organics

Method No.	Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
		1,2-Dibromo-3-chloropropane	<20	80-120	05
		Dibromomethane	<20	80-120	95 06
		1,2-Dichlorobenzene			95 05
			<20	80-120	95
		1,3-Dichlorobenzene	<20	80-120	95
		1,4-Dichlorobenzene	<20	80-120	95
		Dichlorodifluoromethane	<20	80-120	95
		1,1-Dichloroethane	<20	80-120	95
		1,2-Dichloroethane	<20	80-120	95
		1,1-Dichloroethene	<20	80-120	95
		c-1,2-Dichloroethene	<20	80-120	95
		t-1,2-Dichloroethene	<20	80-120	95
		1,2-Dichloropropane	<20	80-120	95
		2,2-Dichloropropane	<20	80-120	95
		1,3-Dichloropropane	<20	80-120	95
		c-1,3-Dichloropropene	<20	80-120	95
		t-1,3-Dichloropropene	<20	80-120	95
		1,1-Dichloropropene	<20	80-120	95
		Ethylbenzene	<20	80-120	95
		Hexachlorobutadiene	<20	80-120	95
		Isopropylbenzene	<20	80-120	95
		4-Isopropyltoluene	<20	80-120	95
		Methylene Chloride	<20	80-120	95
		Naphthalene	<20	80-120	95
		n-Propylbenzene	<20	80-120	95
		1,1,2,2-Tetrachloroethane	<20	80-120	95
		1,1,1,2-Tetrachloroethane	₹20	80-120	95
		Tetrachloroethene	<20	80-120	95
		Toluene	<20	80-120	95
		1,2,3-Trichlorobenzene	<20	80-120	95
		1,2,4-Trichlorobenzene	<20	80-120	95
		1,1,1-Trichloroethane	<20	80-120	95 95
		1,1,2-Trichloroethane	<20	80-120	95 95
		Trichloroethene	<20	80-120	95 95
		Trichlorofluoromethane	₹20	80-120	95 95
		1,2,3-Trichloropropane	<20	80-120	95
		1,2,4-Trimethylbenzene	<20	80-120	95 95
		1,3,5-Trimethylbenzene	₹20	80-120	95 95
		Styrene		80-120	95 95
		Vinyl Chloride	<20		95
		•	<20	80-120	95 05
		o-Xylene	<20	80-120	95 05
		m,p-Xylenes	<20	80-120	95
EPA 601	SW	Bromodichloromethane	<25	70-130	95
	GW	Bromoform	<25	70-130	95
	EFF	Bromomethane	<25	70-130	95
		Carbon Tetrachloride	<25	70-130	95

Table 1 Data Quality Objectives - Organics

Method No	. <u>Matrix</u>	Analyte/Component	Precision % RPD	Accuracy %R	Completeness %
		Chlorobenzene	<25	70-130	05
		Chloroethane			95 05
			<25 <25	70-130	95 95
		2-Chloroethylvinyl ether Chloroform	<25	70-130	9 5
		Chloromethane	<25	70-130	95 05
			<25	70-130	95
		Dibromochloromethane	<25	70-130	95
		1,2-Dichlorobenzene	₹ 25	70-130	95
		1,3-Dichlorobenzene	<25 <25	70-130	95
		1,4-Dichlorobenzene	< <u>/</u>	70-130	95
		Dichlorodifluoromethane	<25	70-130	95
		1,1-Dichloroethane	25 25	70-130	95
		1,2-Dichloroethane	<23	70-130	95
		1,1-Dichloroethene	<25	70-130	95
		t-1,2-Dichloroethene	<25	7 0-130	95
		1,2-Dichloropropane	<25	70-130	95
		c-1,3-Dichloropropene	<25	70-130	95
		Methylene Chloride	<25	70-130	95
		1,1,2,2-Tetrachloroethane	<25	70-130	95
		Tetrachloroethene	<25	70-130	95
		1,1,1-Trichloroethane	<25	70-130	95
		1,1,2-Trichloroethane	<25	70-130	95
		Trichloroethene	<25	70-130	95
		Trichlorofluoromethane	₹25	70-130	95
		Vinyl Chloride	<25	70-130	95 95
EPA 602	HW	_			
EFA 002	LIW	Benzene	<25	70-130	95
		Chlorobenzene	<25	70-130	95
		2-Chloroethylvinyl ether	<25	70-130	95
		1,2-Dichlorobenzene	<25	70-130	95
		1,3-Dichlorobenzene	<25	70-130	95
		1,4-Dichlorobenzene	<25	70-130	95
		1,1-Dichloroethene	<25	70-130	95
		t-1,2-Dichloroethene	<25	70-130	95
		c-1,3-Dichloropropene	<25	70-130	95
		t-1,3-Dichloropropene	<25	70-130	95
		Ethylbenzene	<25	70 -130	95
		Tetrachloroethene	<25	70-130	95
		Toluene	<25	70-130	95
		Trichloroethene	<25	70-130	95
		Vinyl Chloride	<25 <25 <25	70-130	95
		o-Xylene	<25	70-130	95
		m-Xylene	<25	70-130	95
		p-Xylene	<25	70-130	· 95
		-			,,
EPA 602	S	Benzene	<25	70-130	95
	SED	Chlorobenzene	₹25	70-130	95
			~~~	10.130	7.5

Table 1
Data Quality Objectives - Organics

Method No	. <u>Matrix</u>	Analyte/Component	Precision % RPD	n Accuracy <u>%R</u>	Completeness <u>%</u>
	sw	2-Chloroethylvinyl ether	<25	70-130	95
	GW	1,2-Dichlorobenzene	<25	70-130	95
	EFF	1,3-Dichlorobenzene	<25	70-130	95
		1,4-Dichlorobenzene	<ul><li>25</li><li>25</li><li>25</li></ul>	70-130	95
		1,1-Dichloroethene	25	70-130	
		t-1,2-Dichloroethene	-25	70-130	95 05
-		c-1,3-Dichloropropene	25		95 05
		t-1,3-Dichloropropene	থ্য থ্য থ্য	70-130	95 05
		Ethylbenzene	~~; ~~	70-130	95
		Tetrachloroethene		70-130	95
		Toluene	<25	70-130	95
		Trichloroethene	<25	70-130	95
			<25	70-130	95
		Vinyl Chloride	<25	70-130	95
		o-Xylene	<25	<b>70</b> -130	95
		m-Xylene	<25	70-130	95
		p-Xylene	<25	<b>70</b> -130	95
EPA 8280	S	2,3,7,8-TCDD	<30.9	80.2-109	95
EPA 8280	W	2,3,7,8-TCDD	<30.9	80.2-109	95
EPA 8270	SW	Acenaphthene	<31	23.5-72.5	95
	GW	Acenaphthylene	NA	NA	
	SED	Acetophenone	NA	NA	
	S	2-Acetylaminofluorene	NA	NA	
	EFF	Aldrin	NA	NA	
	HW	Aniline	NA	NA	
		Anthracene	NA	NA	
		Benzo(a)anthracene	NA	NA	
		Benzo(a)pyrene	NA	NA	
		Benzo(b)fluoranthene	NA	NA	
		Benzo(g,h,i)perylene	NA	NA	
		Benzo(k)fluoranthene	NA	NA	
		Benzyl alcohol	NA	NA	
		Benzyl Butyl phthalate	NA	NA	
		α-BHC	NA	NA NA	
		в-внс	NA	NA NA	
		δ-BHC	NA		***
		y-BHC		NA	
			NA	NA	
		bis(2-chloroethoxy)methane	NA	NA	
		bis(2-chloroethyl)ether	NA	NA	
		bis(2-chloroisopropyi)ether	NA	NA	
		bis(2-ethylhexyl)phthalate	NA	NA	
		4-Bromophenyl phenyl ether	NA	NA	
		4-Chloroaniline	NA	NA	
		Chlorobenzilate	NA	NA	

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Table 1
Data Quality Objectives - Organics

		Precision	Acmesey (	Completeness
Method No. Matrix	Analyte/Component	% RPD	%R	2011pieteness <u>%</u>
THE PARTY AND ADDRESS OF	1 mm 1 mm Component	./Y_1S1_12	74.15	_70_
	4-Chloro-3-methyl phenol	<42	22-147	95
	2-Chloronaphthalene	NA	NA	
	2-Chlorophenol	<40	23-134	95
	4-Chlorophenyl phenyl ether	NA	NA	
	Chrysene	NA	NA	
	4,4'-DDD	NA	NA	***
•	4,4'-DDE	NA	NA	
	4,4'-DDT	NA	NA	***
	Diallate	NA	NA	
	Dibenzo(a,h)anthracene	NA	NA	
	Dibenzofuran	NA	NA	
	1,2-Dichlorobenzene	NA	NA	
•	1,3-Dichlorobenzene	NA	NA	
	1,4-Dichlorobenzene	<28	10-62	95
	3.3'-Dichlorobenzidene	NA	NA	
	2,4-Dichlorophenol	NA	NA	
	2,6-Dichlorophenol	NA	NA	
	Dieldrin	NA	NA	
	Diethylphthalate	NA	NA	***
	Dimethoate	NA	NA	
	p-Dimethylaminoazobenzene	NA	NA	
	2,4-dimethylphenol	NA	NA	
	α,α-Dimethylphenyl ethylamine	NA	NA	
	Dimethylphthalate	NA	NA	
	Di-n-butyl phthalate	NA	NA	
	Di-n-octyl phthalate	NA	NA	
	1,3-Dinitrobenzene	NA	NA	
	2,4-Dinitrotoluene		19.5-68.5	95
	2,6-Dinitrotoluene	NA	NA	
	Dinoseb	NA	NA	
	Diphenylamine Endosulfan I	NA	NA	700
	Endosulfan II	NA	NA	
	Endosulfan Sulfate	NA NA	NA NA	
	Endrin	NA NA	NA NA	
	Endrin Aldehyde	NA NA	NA NA	
	Ethyl methanesulfonate	NA NA	NA NA	
	Famphur	NA NA	NA NA	
	Fluoranthene	NA NA	NA NA	
	Fluorene	NA NA	NA	
	Heptachlor	NA NA	NA NA	
	Heptachlor Epoxide	NA NA	NA NA	
	Hexachlorobenzene	NA NA	NA NA	
	Hexachlorobutadiene	NA NA	NA Na	
	Hexachlorocyclopentadiene	NA NA	NA NA	
	Hexachloroethane	NA Na	NA NA	
	TACABOTHOLOGUIANG	NA	NA	

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Table 1
Data Quality Objectives - Organics

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy %R	Completeness %
	Hexachloropropene	NA	NA	
	Indeno(1,2,3-cd)pyrene	NA	NA NA	<del></del>
	Isodrin	NA NA		
	Isophorone		NA	_
	Isosafrole	NA NA	NA NA	
	Kepone	NA NA	NA	<del></del>
	Methapyrilene	NA	NA	
		NA	NA	****
	3-Methylcholanthrene	NA	NA	
	2-Methyl-4,6-dinitrophenol	NA	NA	
	Methyl methane sulfonate	NA	NA	
	2-Methylnaphthalene	NA	NA	
	Methyl Parathion	NA	NA	
	2-Methylphenol	NA	NA	
	4-Methylphenol	NA	NA	
	Naphthalene	NA	NA	
	1,4-Nathoquinone	NA	NA	
	1-Naphthylamine	NA	NA	
	2-Naphthylamine	NA	NA	
	2-Nitroaniline	NA	NA	
	3-Nitroaniline	NA	NA	
	4-Nitroaniline	NA	NA	
	Nitrobenzene	NA	NA	
	2-Nitrophenol	NA	NA	
	4-Nitrophenol	<50	D-132	95
	Nitroquinoline-1-oxide	NA	NA	
	n-Nitrosodiburylamine	NA	NA	
	n-Nitrosodimethylamine	NA	NA	
	n-Nitrosomethylethylamine	NA	NA	
	n-Nitrosodi-n-propylamine	<38	D-115	95
	n-Nitrosodiphenylamine	NA	NA	
	n-Nitrosomorpholine	NA ·	NA	
•	n-Nitrosopiperidine	NA	NA	
	n-Nitrosopyrrolidine	NA	NA	
	5-Nitro-o-toluidine	NA	NA	
	Pentachlorobenzene	NA	NA	
	Pentachloronitrobenzene	NA	NA	
	Pentachiorophenol	<50	14-176	95
	Phenacetin	NA	NA	
	Phenanthrene	NA	NA	
	Phenol	<42	5-112	95
	1,4-Phenylenediamine	NA	NA	***
	Phorate	NA	NA	
	2-Picoline	NA	NA	
	Pronamide	NA	NA	
	Pyrene	<31	13-63.5	95
	Pyridine	NA	NA	
	,		4 14 4	

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Table 1 Data Quality Objectives - Organics

Method No	. <u>Matrix</u>	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
		Safrole	NA	NA	
		1,2,4,5-Tetrachlorobenzene	NA NA	NA NA	<del></del>
		2,3,4,6-Tetrachlorophenol	NA NA	NA NA	***
		Thionazine	NA NA	NA NA	
		o-Toluidine	NA NA	NA NA	
		1,2,4-Trichlorobenzene	<b>₹28</b>	22-71	95
		2,4,5-Trichlorophenol	NA	NA	
		2,4,6-Trichlorophenol	NA NA		
		2,4,0-Tricmolophenol		NA NA	
		o,o,o-Triethylphosphorothioate	NA	NA	
		1,3,5-Trinitrobenzene	NA	NA	
EPA 625	SW	Acenaphthene	NA	47-145	95
	GW	Acenaphthylene	NA	33-145	95
	EFF	Aldrin	NA	D-166	95
		Anthracene	NA	27-133	95
		Benzo(a)anthracene	NA	33-143	95
		Benzo(a)pyrene	NA	17-163	95
		Benzo(b)fluoranthene	NA	24-159	95
		Benzo(g,h,i)perylene	NA	D-219	95
		Benzo(k)fluoranthene	NA	11-162	95
		Benzyl Butyl phthalate	NA	D-152	95
		в-внс	NA NA	24-149	95 95
		8-BHC	NA	D-110	
		bis(2-chloroethoxy)methane	NA NA		95 05
		bis(2-chloroethyl)ether		33-184	95 05
		bis(2 chloroisoppout)ether	NA NA	12-158	95 05
		bis(2-chloroisopropyl)ether	NA NA	36-166	95
		bis(2-ethylhexyl)phthalate	NA	8-158	95 95
		4-Bromophenyl phenyl ether	ŅA	53-127	95 95
		4-Chloro-3-methyl phenol	NA	22-147	95
		2-Chloronaphthalene	NA	60-188	95
		2-Chlorophenol	NA	23-134	95
		4-Chlorophenyl phenyl ether	NA	25-158	95
		Chrysene	NA	17-168	95
		4,4'-DDD	NA	D-145	95
		4,4'-DDE	NA	4-136	95
•		4,4'-DDT	NA	D-203	95
		Dibenzo(a,h)anthracene	NA	D-227	95
		1,2-Dichlorobenzene	NA	32-129	95
		1,3-Dichlorobenzene	NA	D-172	95
		1,4-Dichlorobenzene	NA	20-124	95
		3,3'-Dichlorobenzidene	NA	D-262	95
		2.4-Dichlorophenol	NA	39-135	95
		Dieldrin	NA	29-136	95
		Diethylphthalate	NA	D-114	95
		2,4-Dimethylphenol	NA	32-119	95 95
		Dimethylphthalate	NA NA	D-112	95 95
			11/1	10-112	90

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Table 1 Data Quality Objectives - Organics

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
	Di-n-butyl phthalate	NA	1-118	95
	Di-n-octyl phthalate	NA	4-146	95
	2,4-Dinitrotoluene	NA	39-139	95
	2,6-Dinitrotoluene	NA	50-158	95
	Endosulfan Sulfate	NA	D-107	95
	Endrin Aldehyde	NA		95
•	Fluoranthene	NA		95
	Fluorene	NA	59-121	95
	Heptachlor	NA	D-192	95
	Heptachlor Epoxide	NA		
	Hexachlorobenzene	NA	D-152	95
	Hexachlorobutadiene	NA	24-116	95
	Hexachloroethane	NA	40-113	95
	Indeno(1,2,3-cd)pyrene	NA	D-171	95
	Isophorone	NA	21-196	95
	2-Methyl-4,6-dinitrophenol	NA	D-181	95
	Naphthalene	NA	21-133	95
	Nitrobenzene	NA	35-180	95
	2-Nitrophenol	NA		95
	4-Nitrophenol	NA	D-132	95
	n-Nitrosodi-n-propylamine	NA	D-230	95
	Pentachlorophenol	NA	14-176	95
	Phenanthrene	NA	54-120	95
	Phenol	NA	5-112	95
	Pyrene	NA		95
	1,2,4-Trichlorobenzene	NA		95
	2,4,6-Trichlorophenol	NA	37-144	95

^{@ -} Limits calculated using in-house data nd - Not Determined

NA - Not Applicable

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Table 2
Data Quality Objectives - Pesticides/PCBs/Herbicides

Method No.	Matrix	Analyte/Component	Precision % RPD	Accuracy %R	Completeness <u>%</u>
EPA 8080	S SED HW	Aldrin  α-BHC  β-BHC  γ-BHC  γ-BHC  Chlordane (technical)  4,4'-DDD  4,4'-DDT  Dieldrin  Endosulfan I  Endosulfan II  Endosulfan Sulfate  Endrin  Endrin Aldehyde  Endrin Ketone  Heptachlor  Heptachlor Epoxide  Methoxychlor  Toxaphene  PCB 1016  PCB 1221  PCB 1232  PCB 1242  PCB 1248  PCB 1254  PCB 1260	S22 NA NA S218 NA NA S218 NA NA S218 NA NA NA NA NA NA NA NA NA NA S2°	40-120 NA NA NA 40-120 NA NA 38-127 52-126 NA NA 56-121 NA NA 40-131 NA NA NA NA NA NA NA	95 
EPA 608	SW GW EFF TCLP	Aldrin  α-BHC  β-BHC  δ-BHC  γ-BHC  Chlordane (technical)  4,4'-DDD  4,4'-DDE  4,4'-DDT  Dieldrin  Endosulfan I  Endosulfan II  Endosulfan Sulfate  Endrin  Heptachlor  Heptachlor  Heptachlor Epoxide  Toxaphene  PCB 1016	S22 NA NA NA S22 NA NA S18 NA NA S21 S20 NA NA NA NA NA	40-120 37-134 17-147 19-140 40-120 45-119 31-141 30-145 38-127 52-126 45-153 D-202 26-144 56-121 40-131 37-142 41-125 50-114	95 95 95 95 95 95 95 95 95 95 95 95

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Table 2 Data Quality Objectives - Pesticides/PCBs/Herbicides

Method No	Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness %
		PCB 1221 PCB 1232	NA NA	15-178 10-215	95 95
		PCB 1242	NA	39-150	95 95
		PCB 1248	NA	38-158	95 95
		PCB 1254	NA	29-131	
		PCB 1260	≤15	26-147	95
EPA 8150	sw	2,4-D	<30	14-151	95
	GW	2,4,5-T	<40	58-107	95
	EFF	2,4,5-TP (Silvex)	<30	<b>57-113</b>	95
EPA 8150	S	2,4-D	<3	100-141	95
	SED	2,4,5-T	<30●	98-116	95
	HW	2,4,5-TP (Silvex)	<46	66-149	95
EPA 8150	TCLP	2,4-D	<33	28-147	95
		2,4,5-T	<40	44-119	95
		2,4,5-TP (Silvex)	<40	37-149	95

^{@ -} Limits calculated using in-house data

nd - Not Determined

NA - Not Applicable

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Table 3
Data Quality Objectives - Metals

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy %R	Completeness %
EPA 200.7 SW EPA 6010 GW DW EFF	Aluminum Antimony Arsenic Barium Beryllium Bismuth Boron Cadmium Calcium Chromium, Trivalent Cobalt Copper Iridium Iron Lead Lithium Magnesium Manganese Molybdenum Nickel Palladium Phosphorous Platinum Potassium Selenium Silica Silver Sodium Strontium Sulfur Thallium Tin Titanium Uranium Vanadium Zinc	<b>3000000000000000000000000000000000000</b>	75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125	95 95 95 95 95 95 95 95 95 95 95 95 95 9
EPA 200.7 S EPA 6010 SED HW	Aluminum Antimony Arsenic Barium Beryllium Bismuth Boron Cadmium	\(\alpha\)	75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125	95 95 95 95 95 95 95

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Table 3
Data Quality Objectives - Metals

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy Con <u>%R</u>	pleteness <u>%</u>
EPA 245.1 SW SA GW EFF, SED HW		QQ QQ QQ QQ QQ QQ QQ QQ QQ QQ QQ QQ QQ	75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125 75-125	95 95 95 95 95 95 95 95 95 95 95 95 95 9
EPA 204.2, DW 206.2, SW 239.2,	Arsenic Antimony	≤20 ≤20	80-120 80-120	95 95
270.2, GW 279.2,	Lead	≤20	80-120	95
7041, 7060,	Selenium	≤20	80-120	95
7421, 7740, 7841	Thallium	≤20	80-120	95

[@] - Limits calculated using in-house data, nd - Not Determined, NA - Not Applicable
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Table 4
Data Quality Objectives - General Inorganics

Method No.	Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
SM 9221C	DW SW GW EFF	Fecal Coliform (MPN)	nd	nd	
SM 9222D	DW SW GW EFF	Fecal Coliform (MF)	nd	nd	_
SM 9221B EPA 9131	DW SW GW EFF	Total Coliform (MPN)	nd	<b>nd</b>	
SM 9222B EPA 9132	DW SW GW EFF	Total Coliform (MF)	nd	nd	
EPA 305.1 SM 2310B (4a)	SW GW EFF	Acidity	nd	nd	
EPA 310.1 SM 2320B	SW GW EFF	Alkalinity	nd	nd	
EPA 405.1 SM 5210	SW GW EFF	BOD - 5 day	nd	nd	
SM 5210B	SW GW EFF	BOD - 5 day Carbonaceous	nd	nd	
EPA 300.0	SW GW EFF S SED HW	Bromide	nd	nd	

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Table 4
Data Quality Objectives - General Inorganics

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
EPA 300.0 SW GW EFF S SED HW	Chloride	≤11	72-136	95
SM SW 4500Cl-B GW EFF	Chloride	nd	nd	
EPA 330.1 SW SM GW 4500Cl-D EFF	Residual Chlorine	<30	70-130	95
EPA 410.4 SW GW EFF	COD	<30	70-130	95
EPA 110.1 SW SM 2120E GW EFF	Color	nd	nd	_
EPA 120.1 SW EPA 9050 GW SM 2510B EFF	Specific Conductance	nd	nd	
EPA 335.3 SW EPA 9012 GW EFF	Cyanide	<30	70-130	95
EPA 9010A S SED HW	Cyanide	<30	70-130	95
EPA 335.1 SW EPA 335.3 GW EPA 9012 EFF	Cyanide amenable to Chlorination	nd	nđ	
EPA 360.1 SW GW EFF	Dissolved Oxygen	nd	nd	
EPA 1010 SW GW EFF	Flash Point	≤30	80-120	95
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Table 4
Data Quality Objectives - General Inorganics

Method No. M	<u>Matrix</u>	Analyte/Component	Precision % RPD	Accuracy Con <u>%R</u>	mpleteness <u>%</u>
EPA 340.2 S SM 4500F- C		Fluoride	≤30	70-130	95
EPA 130.2 C	SW GW EFF	Hardness	≤30	70-130	95
EPA 350.2 C	SW GW EFF	Ammonia as Nitrogen	<20	80-120	95
EPA 351.2 C	SW GW EFF	TKN	nd	nd	
C E S S	SW GW EFF S SED HW	Nitrate	≤13	82-113	95
C F S S	SW GW EFF S SED HW	Nitrite	≤18	89-118	95
	SW GW EFF	Nitrate/Nitrite	nd	nd	
EPA 350.2 C	SW GW EFF	Organic Nitrogen (TKN-NH ₃ )	nd	nd	
(	SW GW EFF	Odor	nd	nď	

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Table 4
Data Quality Objectives - General Inorganics

Method No.	Matrix	Analyte/Component	Precision % RPD	Accuracy (	Completeness <u>%</u>
EPA 413.1 EPA 9070	SW GW EFF	Oil & Grease	≤30	70-130	95
EPA 413.2 EPA 9071	S SED HW	Oil & Grease	≤30	70-130	95
EPA 150.1	SW GW EFF	pH	nd	nd	
EPA 9045A	S SED	pH ·	nd	nd	
EPA 9041A	HW	pH	nd	nd	
EPA 420.2	SW GW EFF	Phenol	≤30	70-130	95
EPA 365.1 EPA 365.2	SW GW EFF	Ortho-phosphate	≤30	70-130	95
EPA 365.1	SW GW EFF	Total Phosphate	<20	80-120	95
EPA SW846 part 7.3	SW GW EFF S SED HW	Reactivity	nd	nd	
EPA 160.1	SW GW EFF	TDS	≤30	70-130	95
EPA 160.5	SW GW EFF	SS	nd	nd	

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Table 4
Data Quality Objectives - General Inorganics

Method No. Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
EPA 160.2 SW GW EFF	TSS	nd	nd	
EPA 160.3 SW GW EFF	TS	≤30	70-130	95
EPA 170.1 SW SM 2550B GW EFF S SED HW	Temperature	nd	nd	
EPA 300.0 SW GW EFF S SED HW	Sulfate	≤43	79-126	95
EPA 376.1 SW EPA 376.2 GW EPA 9030A EFF EPA 9031	Sulfide	≤30	70-130	95
EPA 377.1 SW GW EFF	Sulfite	nd	nd	
EPA 425.1 SW GW EFF	Surfactants	≤30	70-130	95
EPA 180.1 SW GW EFF	Turbidity	nd	nd	
EPA 415.1 SW EPA 9060 GW EFF	TOC	≤20	70-130	95

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# Table 4 Data Quality Objectives - General Inorganics

Method No.	Matrix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
EPA 160.4	SW GW EFF S SED HW	Volatile Residue	nd	nd	

[@] - Limits calculated using in-house data nd - Not Determined NA - Not Applicable

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Table 5
Data Quality Objectives - Radiochemistry

Method No. Ma	auix	Analyte/Component	Precision % RPD	Accuracy <u>%R</u>	Completeness <u>%</u>
EPA 900	W S V AF	Gross Alpha	≤20 See Note 2	75-125	95
EPA 900	W S V AF	Gross Beta	≤20 See Note 2	75-125	95
EPA 906	W S V AF	Tritium	≤20 See Note 2	75-125	95
EPA EERF-C-01 (See Note 1)	W S V AF	Carbon-14	≤20 See Note 2	75-125	95
EPA 905	W S V AF	Strontium 89/90	≤20 See Note 2	75-125	95
DOE HASL 300	W S V AF	Technetium-99	≤20 See Note 2	75-125	95
LANL EM-9 (See Note 1)	W S V AF	Iodine-129	≤20 See Note 2	75-125	95
EPA EERF App B	W S V AF	Radon-222	≤20 See Note 2	75-125	95
EPA 903.1	W V AF	Radium-226	≤20 See Note 2	75-125	95

^{1.} Method developed by EPI in conjunction with Dr. William Burnett of Florida State University

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If sample and sample duplicate concentration is < 5 times the Minimum Detectable Activity (MDA), the %RPD must be ≤100.

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Table 5
Data Quality Objectives - Radiochemistry

Method No. M	latrix	Analyte/Component	Precision <u>% RPD</u>	Accuracy C	Completeness <u>%</u>
EPA 904	W V AF	Radium-228	≤20 See Note 2	75-125	95
EPA 900.1	W S V AF	Total Alpha Radium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Uranium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Americium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Curium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Thorium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Plutonium	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W S V AF	Neptunium	≤20 See Note 2	75-125	95
EPA 901.1	W S V AF	Gamma	≤20 See Note 2	75-125	95

^{1.} Method developed by EPI in conjunction with Dr. William Burnett of Florida State University

If sample and sample duplicate concentration is < 5 times the Minimum Detectable Activity (MDA), the %RPD must be ≤100.

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Table 5 Data Quality Objectives - Radiochemistry

Method No. Ma	atrix	Analyte/Component	Precision % RPD	Accuracy Con	npleteness %
DOE HASL 300 (See Note 1)	W S V AF	Polonium	≤20 See Note 2	75-125	95
EPA 902	W V AF	Iodine-131	≤20 See Note 2	75-125	95
DOE HASL 300 (See Note 1)	W V AF	Lead-210	≤20 See Note 2	75-125	95
DOE RESL P-1-1	W S V AF	Phosphorus-32	≤20 See Note 2	75-125	95
EPA EERF PM-01-1 (See Note 1)	W S V AF	Promethium-147	≤20 See Note 2	75-125	95
DOE RESL Ni-1	W S V AF	Nickel-59	≤20 See Note 2	75-125	95
DOE RESL Ni-1	W S V AF	Nickel-63	≤20 See Note 2	75-125	95
DOE RESL- A-3-1	W S V AF	Radium-223	≤20 See Note 2	75-125	95
DOE RESL- A-3-1	W S V AF	Radium-224	≤20 See Note 2	75-125	95

Method developed by EPI in conjunction with Dr. William Burnett of Florida State University
 If sample and sample duplicate concentration is < 5 times the Minimum Detectable Activity</li> (MDA), the %RPD must be ≤100.

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#### IV. SAMPLING PROCEDURES

Sampling is the process of collecting a representative portion of the environment to learn about the whole environment. A representative sample is one that typifies the rest of the environment. To collect representative samples, one must standardize sampling bias related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Collecting a representative sample and maintaining its integrity until it is analyzed is important because the validity of each analysis begins with the sampling.

# A. Groundwater Sampling Protocol

The procedures described in this section are guidelines utilized by GEL to ensure that representative groundwater samples are collected. Procedures for measurement of the water table depth, total well depth, well evacuation, and sample collection are included.

All data collected and observations made in the field are recorded on the Field Data Information Sheet for Groundwater Sampling which is included as Appendix 3. Prior to the initiation of any activities at each well site, all sampling personnel must put on new laboratory quality PVC gloves. These gloves are replaced, as necessary, during the well evacuation and sampling process and are always changed between wells. Prior to the collection of any data at a particular well, the surface integrity of the well is checked and any problems noted. When the well cap is removed, any odors that are detected are noted on the Field Data Information Sheet.

#### Water Level Measurement

Prior to the evacuation of any monitoring well, the depth to the water table is determined with the use of a Well WizardTM Model 6000, or equivalent, portable electronic water level indicator. The electronic water level indicator uses a sensitive circuitry to activate a buzzer when electrical continuity is made at the probe. The sensitivity is set so that water with a specific conductance greater than approximately one µmhos/cm closes the circuit.

The water level in wells is measured with the electronic water level indicator by turning the instrument on and slowly lowering the instrument probe into the well until the light and buzzer are activated. The distance to the water is determined by measuring the distance from the measuring point on the well casing to the water level. The instrument is calibrated in 0.1 foot increments. However, all measurements will be made and interpolated to the nearest 0.01 foot.

The instrument is cleaned according to the "Field Cleaning Procedure" that is included in this section. The instrument is shut off following each use.

# Total Depth Measurement

The total depth of all monitoring wells, except those equipped with dedicated pump devices, is measured on a semi-annual basis to determine if excessive siltation has occurred in the well casing. The total depth measurement is used in the calculation of the volume of

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water standing in the well casing. The following procedure is used for measurement of the total well depth:

- 1. The Well WizardTM Model 6000 (or equivalent) is slowly lowered into the well until the bottom is encountered with the steel probe.
- 2. The calibrated tape is raised and lowered until the probe is resting on the well bottom and tension is maintained.
- 3. The calibrated tape is read at the point which intersects the established measuring point.
- 4. This distance is recorded to the nearest 0.01 foot.
- 5. A factor of 0.35 feet (dependent on model) is added to the measured distance. This factor compensates for the length of the probe tip not included in the calibration of the tape.
- 6. The resulting measurement is recorded as the Total Well Depth on the Field Data Information Sheet.
- 7. The Well WizardTM Model 6000 (or equivalent) is then cleaned according to the Field Cleaning Procedure.

#### Detection Of Immiscible Levels

For the detection of immiscible liquids, the ORSTM Model 1068013 Interface Probe, or equivalent, is slowly lowered into the well until an oscillating or continuous alarm sounds and light is activated. When the probe contacts liquid, the visual/audible alarm on the reel will be activated. An oscillating alarm indicates water, a continuous alarm indicates hydrocarbon. To determine the exact thickness of a hydrocarbon layer, the probe should be slowly lowered to the air/hydrocarbon interface until the alarm is activated. With the probe at the exact point where the alarm comes on, read the numbers on the tape to determine the distance from the notch in the top of the wellhead tape guide to the air/hydrocarbon interface. Subtract 2/10' (6.1 cm) from the reading to compensate for the height of the wellhead tape guide. Next lower the probe through the hydrocarbon layer and well into the water. An oscillating alarm will be obtained. The probe is then raised slowly to the hydrocarbon/water interface until the point is reached where the alarm changes from oscillating to continuous. The thickness of the hydrocarbon layer is determined by subtracting the first reading from the second reading. The ORSTM Model 1068013 Interface Probe is then cleaned according to the Field Cleaning Procedure.

#### Well Evacuation

The purpose of the well evacuation procedure is to remove any stagnant water from the well prior to the collection of samples for analysis. The procedure ensures that adequate mixing has taken place in the well column and that the water being removed for analysis is representative of the water immediately surrounding the well. For wells which recover immediately, a minimum of three casing volumes is removed prior to sampling. Indicator parameters (pH, Specific Conductance, and Temperature) are tested at certain intervals during the evacuation process to ensure representative water quality. Additional water is removed from the well until three consecutive stable measurements of pH and specific conductance are achieved. Wells which can be evacuated to dryness prior to reaching the three casing volume standard will be measured for pH and specific conductivity and sampled as soon as a sufficient volume of water has entered the well. Additional criteria concerning the sampling procedures for low yield wells is included later in this section on sampling procedures.

Well evacuation is accomplished using primarily Teflon™ bailers. The sampling line to each well is nylon or polypropylene rope. In some cases, where applicable, diaphragm pumps, hand pumps, and centrifugal pumps are used for evacuation.

The following steps are followed for evacuation of monitoring wells:

- 1. The depth of the water table is subtracted from the total well depth to determine the length of the water column. The volume of water in the column is then converted to gallons of water in the well casing. The casing volume is multiplied by 3 to obtain the minimum (standard) evacuation volume.
- 2. The bailer is lowered just below the top of the water level each time to prevent agitation of solids but ensure adequate mixing in the water column.
- 3. Measurements for pH, specific conductance, and temperature are taken after the evacuation of each well volume. All times, volumes, and measurements are recorded immediately on the Field Data Information Sheet.

# Sampling Procedures for Groundwater

The primary consideration for collecting groundwater samples for analysis is to ensure that the sample is not altered or contaminated during withdrawal from the well, during introduction to the sample container, or during transportation. Special procedures are often necessary for sampling monitoring wells based on the yield of the well. A high yield well is defined as a well that cannot be drawn down by bailing or pumping by hand. A "moderate" yield well can be drawn down; however, it cannot be evacuated to dryness. A low yield well can be evacuated to dryness and requires from a few hours to several days to fully recover.

For moderate to high yield wells, a complete set of new Superfund Certified labeled sample bottles are removed from the cooler and fresh sample is slowly poured into each container. Preservatives are added as necessary, according to the EPA Guidelines for preservatives in 40 CFR, Part 136, Table II. Preservatives and bottle requirements are listed in Appendix 2. The filled bottles are securely placed into the pre-cleaned cooler and a Chain of Custody form is completed for that well. A copy of the chain of custody is found in Appendix 4.

Special procedures are necessary for the collection of samples from low yield wells. The wells are evacuated until completely dry and then as the well begins to recover, samples are collected. When full recovery exceeds three hours, the remaining samples are collected in order of their volatility as soon as sufficient volume is available for a sample of each parameter. Samples collected for volatile organics, total organic carbon, and halogens are handled with extreme care to prevent aeration. No headspace is left in sample containers to minimize the possibility of volatization of organics. Notations are made in the field and in the lab of any headspace remaining in the container. Samples for bacteriological analysis are collected in pre-autoclaved containers to which a preservative has been added. Once the container is filled, preservative(s) is added when necessary, the sample container is placed into the cooler, and chain of custody documentation is initiated.

# B. Surface Water Sampling Protocol

Many factors are involved in selecting an appropriate sampling site for the collection of surface water samples, including:

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- Accessibility
- Flow
- Mixing
- Other Physical Characteristics of the Water Body
- Point and Diffuse Sources of Contamination
- Personnel and Equipment available to conduct the study

In case of dissolved constituents, dispersion depends on the vertical and lateral mixing within the cross section of a body of water.

Most monitoring studies of streams and lakes aim to assess the effects of overland runoff, groundwater inflow, or waste disposal into waterways. Such studies can usually be tied to the physiographic features of the area under consideration. In most cases, the quality of the water flowing into a lake or past a particular point along a stream can be related directly to inputs within the drainage basin, including the entire drainage area upstream of a selected point. This is also true for marshes and tide influenced waterways.

#### Sampling Procedures for Rivers and Streams

The procedures described in this section are intended to give a guideline to ensure that representative surface water samples are collected.

Procedures for collection of samples from streams and rivers vary from the simplest of hand sampling procedures at a single point to more sophisticated multi-point sampling techniques. Ideally, samples are collected that represent both dissolved and suspended constituents, and vertical and horizontal distributions.

All stream samples are collected by depth-integrating methods using either a handheld or a cable-and-reel sampler whenever practical. In shallow streams where the depths are insufficient for true depth integration, grab samples collected at one or more verticals across the stream are appropriate.

#### Grab Sampling

A grab sample is obtained using a new set of Superfund Certified sample containers. The sampling site is identified on the Field Data Information Sheet and the Chain of Custody. The container cap is removed and the container is then held at the base with one hand and the container mouth is placed quickly down into the water to avoid introducing floating materials. The mouth of the container is positioned into the current away from the hand of the collector and away from the side of the sampling platform or boat. The sampling depth is usually 15 to 30 cm (6 to 12 inches) below the water surface. If the water body is static, an artificial current is created by moving the container horizontally in the direction that is pointed away from the sampler. The container is tipped slightly upwards to allow air to exit and the container to fill. The container is tightly capped and labeled appropriately.

#### Depth Sampling

Several additional devices are needed for the collection of depth integrated samples for lakes, reservoirs, estuaries, and oceans. These depth samplers require lowering the

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sampling device and/or container to the desired depth; opening, filling, and closing the device and/or container; and returning the device and/or container to the surface.

# C. Sediment Sampling Protocol

A wide variety of samplers and procedures have been developed and are commonly used for the sampling of sediments.

In order to obtain satisfactory samples, the sediment samplers enclose a volume of the bed material and then isolate the sample from water currents while the sampler is being lifted to the surface of the stream. The ease by which the sample can be transferred to a suitable container is important.

The selection of the best procedure for any given situation is one of the most important decisions to make in sediment work. Besides deciding on the proper sampler and sampling depth, judgments are made on the number and pattern of sample collecting and on whether to composite the samples prior to analyses.

The procedures included under this heading are used as a guideline to ensure that representative sediment samples are collected. Record all data collected in the field on the Field Data Information Sheet.

# Sampling Procedures for Sediment in Rivers and Streams

Surface bed material is the sediment in the topmost layers of the stream bed. The procedures for sampling this material depend on the depth of flow, the size of the particles, and how the sample analysis results are to be used.

In most circumstances, a number of samples are collected at or near a cross section to characterize the bed material. The samples are sometimes combined into a single composite sample to provide a mean value(s) for the variable(s) under study, provided that the material is fairly homogeneous. Samples of dissimilar character should not be combined for a single analysis.

All soil samples are stored according to the guidelines set forth in the document EPA/CE-81-1 (Procedures for Handling and Chemical Analysis of Sediment and Water Samples, US EPA and Corps of Engineers Manual).

#### Shallow Samples

Samplers for obtaining material moderately close to the bed surface generally fall into one of the following types:

- Drag bucket or scoop
- Grab bucket or clamshell
- Vertical pipe or core
- Rotating bucket

The drag bucket or scoop and the grab bucket or clamshell types are not usually recommended for sampling the bed of flowing streams. The characteristics of the drag bucket are such that some of the sample material may wash away, and the clamshell does

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not always close properly if the bed contains gravel. In situations where these samplers are used, a special effort must be made to determine the representativeness of the samples.

The vertical-pipe or core samplers are essentially tubes which are forced into the stream bed. Penetration in fine-grained sediment is easy; however, penetration in sand usually is limited to about 1 meter or less. During retrieval, the sample is retained inside the cylinder by a partial vacuum formed above the sample and/or by a core retainer at the lower end. In many cases, clear tubes are used in order to characterize vertical strata.

# D. Soil Sampling Protocol

There are two primary portions of the soil that are important to the environmental scientist. The surface layer (0-15 cm) reflects the deposition of recently deposited pollutants. Pollutants that have been deposited by liquid spills, or by long term deposition of water soluble materials may be found at depths ranging up to several meters. Plumes emanating from hazardous waste units or leaking storage tanks may be found at considerable depths. The methods for sampling each of these are slightly different; however, all make use of one of two basic techniques. Samples are either collected with some form of core sampling or auger device, or by use of excavations or trenches. In the latter case, the samples are cut from the soil mass with spades or short punches. The American Society for Testing and Materials (ASTM) has developed a number of methods that have direct application to soil sampling. These often need to be modified slightly to meet the needs of the environmental scientist that requires samples for chemical analyses since the ASTM Methods are designed primarily for engineering tests. The techniques that are utilized should be closely coordinated with the analytical laboratory in order to meet the specific requirements of the analytical methods used, including sample preservation and storage.

# Surface Sampling Procedures for Soils

Surface soil sampling is divided into two main categories: the upper 15 cm and the upper meter. The very shallow pollution, such as that found downwind from an air source or at sites of recent spills of relatively insoluble chemicals, may be sampled using one of the procedures discussed in this section. The deeper pollutants found in the top meter are the more soluble, recent pollutants or those that were deposited on the surface a number of years ago. These have begun to move downward into the deeper soil layers. One of the surface procedures discussed in this section is used in those cases.

# Sampling with a Soil Punch

A number of studies of surface soils have made the use of a punch or thin-walled steel tube that comes in various lengths to extract short cores from the soil. The tube is driven into the soil with a mallet, the core and the tube are extracted, and the soil is pushed out of the tube into a stainless steel mixing bowl and composited with other cores, if necessary. These samples are then transferred into proper sample containers.

#### Shallow Subsurface Sampling

Precipitation may move surface pollutants into the lower soil horizons or move them away from the point of deposition by surface runoff. Sampling pollutants that have moved into the lower soil horizons require the use of a device that will extract a longer core

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than can be obtained with the short probes or punches. Three basic methods are used for sampling these deeper soils:

- Soil probes or soil augers
- Power driven corers
- Trenching

The soil probe collects intact, relatively undisturbed, soil cores. The auger collects a "disturbed" sample in approximately the same increments as the probe. Power augers can use split spoon samplers to extract cores up to 60 cm long. With special attachments, longer cores can be obtained with the power auger if necessary.

#### Soil Probes and Hand Augers

Two standard tools used in soil sampling are the soil probe (often called a Kingtube) and the hand auger. These tools are designed to acquire samples from the upper two meters of the soil profile. The soil probe is a stainless steel or brass tube that is sharpened on one end and fitted with a long, T-shaped handle. These tubes are usually approximately 2.5 cm inside diameter although larger tubes may be obtained. The cores collected by the tube sampler or soil probe are considered to be "undisturbed" samples. The tube is pushed into the soil in approximately 20 to 30 cm increments. The soil core is then removed from the probe and placed in either the sample container or in a mixing bowl for compositing.

The hand auger can also be used to obtain soil samples. The samples are "disturbed" using this procedure and should not be used when it is necessary to have a core to examine or when very fine detail is of interest. The auger is twisted into the soil and then extracted. Because of the length of the auger and the force required to pull the soil free, only 20 to 30 cm maximum length can be extracted at one time. In very tight clays, it may be necessary to limit the length of each pull. Precautions must be taken to minimize the possibilities for cross contamination.

#### Trenching

This procedure is used to carefully remove sections of soil during studies where a detailed examination of pollutant migration patterns and detailed soil structure are required. It is perhaps the least cost effective sampling method because of the relatively high cost of excavating the trench from which the samples are collected. It is used only in those cases where detailed information is required.

A trench may be from 1 meter to 4 meters deep and is typically done using a backhoe or other trenching device. The samples are taken from the sides of the pit using the soil punch or a trowel.

# E. Field Quality Control

A strict program of Field Quality Control is maintained by GEL to ensure that sample integrity is maintained during sample collection and transport to the laboratory. In addition, all field equipment and instruments are properly maintained and calibrated to prevent sample bias and ensure that field measurements are accurate.

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## Sample Labels

Sample labels are necessary to prevent misidentification of samples. Gummed paper labels or tags are adequate and include the following information:

- Sample Identification Number
- Name of the Collector
- Date and Time of Collection
- Place of Collection
- Well Number, where applicable
- Parameters for Analysis

Labels are affixed to sample containers prior to or at the time of sampling. The labels are filled out at the time of collection.

### Field Cleaning Procedures

All field equipment and instrumentation is cleaned at the laboratory according to standard laboratory procedures as soon as possible after return after each use. Field equipment and instrumentation include:

- Augers
- Bailers
- Coolers
- Pumps
- pH Meters
- Specific Conductivity Meters
- Tape measures
- Temporary sample containers

Field equipment and instrumentation which must be used on multiple wells are cleaned in the field according to the following field cleaning procedure:

- 1. Flush item thoroughly with 5% phosphate-free laboratory detergent solution
- 2. Rinse item at least two times with potable water
- 3. Rinse item at least one time with deionized water
- 4. Return item to its protective case or wrap in aluminum foil or plastic

For organics sampling, a solvent rinse between the potable water and the deionized water rinses must be included. Nanograde isopropanol is commonly used.

For more specialized field cleaning and decontamination procedures, refer to EPA document "Standard Operating Procedures and Quality Assurance Manual (February 1, 1991), Appendix B, Standard Cleaning Procedures".

#### Field Log Book

All information pertinent to a field survey or sampling is recorded in a logbook. Entries in the logbook typically include the following:

- Purpose of sampling, i.e. surveillance, contract number
- Location of sampling point

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- Description of sampling point and sampling methodology
- Date and time of collection
- Sample Identification Number(s)
- Sample distribution and how transported, i.e. name of laboratory, UPS, etc.
- Maps of the sampling site
- Field observations
- Any field measurements made, i.e. pH, Specific Conductance, etc.

#### Field Data Information Sheet

All pertinent field information is recorded on the appropriate Field Data Information Sheet at the time of collection. This information includes:

- Date of Sampling
- Name of the Collector
- Sampling Location
- Sample Identification
- Method of Sampling
- All Other Pertinent Field Data and Conditions

In addition, information for monitoring wells includes:

- Monitoring Well Number
- Casing Diameter
- Material of Construction
- Well Integrity
- Well Protection
- Measuring Point Elevation
- Total Well Depth
- Depth to Groundwater
- Volume of Water in Casing
- Method of Evacuation
- Total Volume of Water Evacuated
- Field Analyses with Time and Volume Evacuated

The field data sheets are reviewed upon arrival to the laboratory. They are cross-checked against labeled sample containers to ensure that all of the information is correct.

#### Sample Transportation and Chain of Custody

The transportation of samples from the time they are collected to the laboratory is an integral part of the sampling program. Transportation of samples and maintaining the chain of custody are discussed in detail in Section V of this Quality Assurance Plan under the heading of Sample Custody.

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#### V. SAMPLE CUSTODY

Sample custody is essential for defensible environmental data. Chain of custody procedures provide assurance of traceability and integrity for samples needed for legal purposes. Sample custody is a planned mechanism for tracking a sample from the collection of the sample in the field through the disposal of the sample.

A sample is under custody if:

- It is in your physical possession; or
- It is in your view, after being in your possession; or
- It is locked up after being in your possession; or
- It is in a designated secure area.

Samples are collected in proper containers and labeled to assure sample tracking. The chain of custody form is initiated in the field at sample collection by field personnel. This includes such information as the client name and parameters for which samples are being collected. The sample locations are also noted on the chain of custody. Other information on the form includes the date and time of collection; the sampling technique; filtering procedures; sample preservation method, including reagents and whether preservation occurred in the laboratory or in the field; and any comments or remarks that may be useful in the analytical work or data interpretation that will follow. Additional space on the form permits each individual to sign for the sample as its custody is relinquished from one individual and received by the next person.

After sampling, chain of custody is maintained as field personnel relinquish the samples in properly prepared, sealed shipping containers to a company courier or third party delivery service.

When sampling is handled by the client rather than GEL personnel, it is the responsibility of the client to maintain field quality control and chain of custody until the samples arrive at GEL. In these instances, GEL can only verify that proper preservation and chain of custody have been maintained from the time that the sample arrived at the laboratory. If there is reason to suspect that chain of custody has been broken, GEL will notify the client before any analysis is performed.

#### Sample Receiving

Samples are received at GEL in a central sample receiving area. At the point of delivery, the laboratory sample custodian, also called the log-in clerk, signs for incoming field samples and maintains the shipping documents. The log-in clerk verifies the data entered on the incoming documents and chain of custody and that the temperature and pH of the sample(s) are within the required range. Log-in personnel enter the sample information into the database of the LIMS.

Login personnel follow GEL SOP GL-SR-E-001 for Sample Receipt, Login, and Storage. The SOP explains the process of recording client identification information and assigning sample identification numbers, test codes for analyses, and analysis turn-around-time. It also describes how samples are cross-checked against documentation, how to resolve problems, and sample transfer. An automatic label generator prints a label for each

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sample container. This label contains the sample number as well as other important information for sample analysis. The sample is placed in storage until analysis. All of the sample storage units are in secured areas, and only authorized personnel have access to these areas.

Daily log sheets of received samples are generated. This sheet is signed by the login clerk and placed in a bound logbook kept in Sample Receiving. An example of a daily log is included as Appendix 5.

Samples are removed from cold or ambient storage the day after release of the completed sample and placed in the Residue Area. The Residue Area is located in a secure area at the rear of the building in High Bay. Samples are stored until disposed or returned to the client.

# Internal Chain of Custody

The LIMS Internal Chain of Custody System assigns, tracks, and generates reports on the chain of custody of sample containers and their extracts as they move through the laboratory. Barcode labels are generated when samples arrive in Sample Receiving and when extracts are created in the laboratory. These sample containers and extracts are then scanned using barcode scanners as they move through the laboratory with the user's name, location, date, and time scanned attached to each record.

# Radioactive Samples

While similar custody tracking procedures are used for all samples, radioactive samples are handled following SOP GL-EPI-E-S007 for Receiving of Radioactive Samples and SOP GL-EPI-E-S005 for Radioactive Waste Handling. GEL is limited by license in the amount of radioactive material that may be in the laboratory at any given time. Before GEL accepts radioactive samples, the client must provide prior notice of shipment of radioactive samples and the level of activity for the sample to be shipped must be declared. Upon the arrival of radioactive samples at the sample receiving location, all radioactive samples are handled only by designated personnel who have been trained in the proper handling of radioactive materials.

Prior to log-in, all sample containers are monitored for levels of radioactivity. If a reading is greater than 2mR/Hr, the sample is not accepted. It is immediately repacked and shipped back to the client.

When GEL accepts the shipment of radioactive samples, lab numbers are assigned to each sample and a Radioactive Shipment Inventory Sheet is originated. This form is provided as Appendix 6. The radioactivity level and the amount of sample received must be entered on the forms.

Radioactive samples are maintained in a specially designated walk-in cooler. All sample and shipping containers are tagged to denote the presence of radioactivity.

An inventory of the amount of sample taken for analysis and the amount of sample and/or waste returned following analysis must be noted on the Radioactive Shipment Inventory Sheet. All samples, wastes, reagents contacting samples, and trash associated with radioactive samples, including gloves and aprons, are retained and returned to the client, or transferred to a licensed disposal facility.

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Prior to return of the samples, the level of radioactivity for each shipment of radioactive samples to be returned must be noted on the Radioactive Shipment Inventory Sheet. The Radioactive Shipment Inventory Sheet and Sample Inventory Sheet are released by the Radiation Protection Officer and filed in GEL's Quality Record System.

# Sample Disposal

Sample disposal follows GEL Laboratory Waste Management Plan and is handled by the Waste Management Technician. Non-radioactive samples are composited by sample matrix into storage drums. When a drum is full, it is analyzed for hazardous waste characterizations prior to disposal. Unless otherwise specified in the contract, samples are composited 15 days after invoicing.

SOP GL-EPI-E-S008 for Shipping of Radioactive Samples and Sample Residues describes the steps necessary to ship radioactive samples and residues to the client.

If samples are to be returned to the client, GEL SOP GL-SR-E-002 for Return of Samples is followed.

#### Facility and System Security

GEL SOP GL-FC-E-001 for Facility Security describes the security system for the building and laboratory. This SOP details the steps necessary for entering and exiting the buildings for employees and visitors. Descriptions of the card reader system and the burglar alarm system are included in the SOP.

LIMS requires passwords to access various levels. Each employee with clearance to log on the system must enter a LIMS identification code, an account number, and an individual password before given clearance to enter the system.

# VI. CALIBRATION PROCEDURES AND FREQUENCY

### Calibration

All instruments are calibrated at the frequency of the instrument's use, stability, and specific method requirements. The calibration procedure for each instrument is described in the appropriate analytical standard operating procedure.

The calibration procedures described in the SOPs are not restated in this QAP but an overview is presented below.

Instrument	Procedure
GC/ECD	Meet acceptance criteria (degradation, retention time stability). Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
GC-FID	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
GC-PID	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
GC/MS	Meet tuning criteria.  Multi-point calibration curve with correlation coefficient ≥0.995.  Verify calibration with check standard periodically.
AA Furnace	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
IC	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard every 20 samples.
ICP	Multi-point (minimum of 2 calibration standards) calibration curve with correlation coefficient ≥0.995.  Verify calibration with check standard periodically.
AA Spectrophotometer	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
Conductivity Meter	4 point calibration curve.
pH Meter (Orion EA 940)	3 point calibration curve with slope ≥92%.
Spectrophoto- meter	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.

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Instrument	Procedure
TOC (DC-80)	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
TOX (Cosa TOX-10)	1 point calibration within 97-103% of true value. Analyze calibration standard every 4 samples. Verify calibration with check standard every 15 samples.
Balance	Annual calibration by outside service technician.  Daily calibration verification using certified weights.
Mercury Analyzer	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
Thermometers	Verify against NIST thermometer annually.
GC/ELCD	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard every 20 samples.
Ion Selective Electode	5 point calibration with slope -54 to -60 mV.
Alpkem Rapid Flow Analyzer (RFA)	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
TRAACS 800	Multi-point calibration curve with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
Infrared Spec- trophotometer	3 point calibration with correlation coefficient ≥0.995.
Turbidimeter	3 point calibration with correlation coefficient ≥0.995. Verify calibration with check standard periodically.
Bomb Calorimeter	10 analyses of benzoic acid. Standard deviation must be ≤6.5 BTU/°C.
Dohrmann DC- 190	1 point calibration analyzed in duplicate. Relative percent difference must be ≤4%.

### Method Detection Limits

Method Detection Limits (MDL's) are established for each analytical method routinely used in the laboratory. MDL's are necessary for demonstrating method proficiency and are determined annually by analysis of a minimum of seven replicates in a specific matrix containing the analyte(s). GEL SOP GL-LB-E-001 for the Determination of Method Detection Limits describes all of the steps necessary to estimate and determine a detection limit.

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#### VII. ANALYTICAL METHODOLOGIES

Analyses are performed at GEL using reference methods by Environmental Protection Agency (EPA), Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC), American Society for Testing and Materials (ASTM), Standard Methods for the Examination of Water and Wastewater (SM), South Carolina Department of Health and Environmental Control (SCDHEC) and Code of Federal Regulations (CFR) Titles 40 and 49.

Radiochemistry analyses are performed at EPI using reference methods by EPA, ASTM, the Department of Energy Environmental Measurements Laboratory (EML), and Los Alamos Health and Environmental Chemistry (LAHEC). In addition to these references, many radiochemistry procedures were developed in conjunction with Florida State University (FSU) under the guidance of Dr. Bill Burnett.

GEL and EPI's Standard Operating Procedures Manuals include procedures for all routine analyses performed by the laboratories. These procedures are step-by-step instructions for sample preparation, analysis, instrument calibration, quality control requirements, and reporting of data.

# A. Organic Chemistry Procedures

Base/Neutral and Acid Extractable Compounds - 40 CFR Part VIII Method 625; EPA SW-846 Method 8270. GEL SOP GL-OA-E-009 for Semivolatile Analysis by Gas Chromatograph/Mass Spectrometer describes the steps necessary to determine semivolatile organic compounds by gas chromatography/mass spectrometry in groundwater, wastewater, TCLP extracts, soil, oil, and miscellaneous matrices using a mass selective detector. Aqueous samples are prepared according to SOP GL-OA-E-016 for Extraction of Semivolatile Base-Neutral/Acid (BNA) Compounds from Groundwater and Wastewater. Soil and sludge samples are prepared according to SOP GL-OA-E-010 for Extraction of Semivolatile Organic Compounds from Soil and Sludge Samples.

BTEX by GC - 40 CFR Part VIII Method 602; EPA SW-846 Method 8020. GEL SOP GL-OA-E-023 for BTEX Volatile Organic Aromatics by Gas Chromatograph and Photoionization and Flame Ionization Detectors describes the steps necessary to determine volatile aromatic organic compounds by gas chromatography in groundwater, wastewater, soil, sludge, oil, and miscellaneous matrices using photoionization and flame ionization detectors.

Chlorinated Herbicides - EPA SW-846 Method 8150. GEL SOP GL-OA-E-011 for Chlorophenoxy Acid Herbicides describes the steps necessary to determine certain chlorophenoxy acid herbicides by gas chromatography in water, soil, and TCLP extracts using an electron capture detector. Aqueous samples are prepared according to SOP GL-OA-E-015 for Extraction of Herbicides from Groundwater and Wastewater. Soil and sludge samples are prepared according to SOP GL-OA-E-027 for Extraction of Herbicide from Soil and Sludge Samples.

Drinking Water Volatiles by Gas Chromatograph/Mass Spectrometer - EPA 600/4-88/039 Method 524.2. GEL SOP GL-OA-E-022 for Volatile Organic Compounds

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by Gas Chromatograph/Mass Spectrometer describes the steps necessary to determine purgeable volatile organic compounds by gas chromatography/mass spectrometry in finished drinking water, raw source water, and drinking water at any treatment stage using a purge and trap system and mass selective detector.

Industrial Solvents by Gas Chromatograph - EPA SW-846 Methods 8000 and 8015. This is a gas chromatographic method for the quantitative determination of high concentrations of certain solvents in waste materials and solids using a flame ionization detector.

Moisture Percentage - EPA SW-846 Method 3550. GEL SOP GL-OA-E-020 for Percent Moisture describes the steps necessary to determine the percent moisture in soil and sludge.

Organochlorine Pesticides and PCBs. 40 CFR Part VIII Method 608; EPA SW-846 Method 8080 - GEL SOP GL-OA-E-017 for Polychlorinated Biphenyls describes the steps necessary to determine polychlorinated biphenyls by gas chromatography in water and soil using an electron capture detector. SOP GL-OA-E-018 for Organochlorine Pesticide describes the steps necessary to determine organochlorine pesticides by gas chromatography in groundwater, wastewater, soil, sludge, TCLP extracts, and miscellaneous matrices using an electron capture detector. Aqueous samples are prepared according to SOP GL-OA-E-013 for Extraction of Semivolatile Pesticides and Polychlorinated Biphenyls (PCB) from Groundwater and Wastewater. Soil and sludge samples are prepared according to SOP GL-OA-E-019 for Extraction of Pesticides and Polychlorinated Biphenyls (PCB) from Soil and Sludge Samples.

Otto Fuel II - Method supplied by US Department of the Navy. GEL SOP GL-OA-E-012 for Determination of Trace Amounts of Otto Fuel II in Water and Soil Samples by GC/ECD describes the steps necessary to determine Otto Fuel II by gas chromatography in water and soil using an electron capture detector.

Polychlorinated Biphenyls in Oil - EPA Method "Analysis of Transformer Oils for Polychlorinated Biphenyls". This is a chromatographic method for quantitatively determining polychlorinated biphenyls (PCBs) in oil.

Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans by GC/MS - EPA SW 846 Method 8280. GEL SOP GL-OA-E-007 for Dioxins and Furans describes the steps necessary to determine dibenzo-p-dioxins and dibenzofurans by gas chromatography/mass spectrometry in groundwater and soil.

Total Petroleum Hydrocarbon by GC - EPA SW-846 Methods 3510 and 3550; California Method of Analysis of Total Petroleum Hydrocarbons. GEL SOP GL-OA-E-003 for Non-Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector describes the steps necessary to determine non-volatile total petroleum hydrocarbons by gas chromatography in water, soil, oil, and sludge using a flame ionization detector. SOP GL-OA-E-004 for Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector describes the steps necessary to determine volatile total petroleum hydrocarbons by gas chromatography in water soil, oil, and sludge using a flame ionization detector.

Volatile Organics by GC - 40 CFR Part VIII Methods 601 and 602; EPA SW-846 Methods 8010 and 8020. GEL SOP GL-OA-E-014 for Volatile Organic Hydrocarbons by GC/ELCD describes the steps necessary to determine volatile halogenated compounds by

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gas chromatography in groundwater, wastewater, soil, and miscellaneous matrices using an electrolytic conductivity detector. SOP GL-OA-E-021 for Volatile Organic Aromatic Compounds by Gas Chromatograph and Photoionization Detector describes the steps necessary to determine volatile aromatic compounds by gas chromatography in water, wastewater, soils, sludge, oils, and miscellaneous matrices using a photoionization detector.

Volatile Organics by GC/MS - 40 CFR Part VIII Method 624; EPA SW-846 Methods 8240 and 8260. GEL SOP GL-OA-E-008 for Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer describes the steps necessary to determine purgeable organic compounds by gas chromatography/mass spectrometry in groundwater, wastewater, TCLP extracts, soil, oil, and miscellaneous matrices using a 0.542mm column, purge and trap system, and mass selective detector. SOP GL-OA-E-026 for Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer describes the steps necessary to determine purgeable organic compounds by gas chromatography/mass spectrometry in groundwater, aqueous sludge, caustic liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filtercake, spent carbons, spent catalysts, soils, and sediments using a 0.25mm column, purge and trap system, and mass selective detector.

#### B. Metals Procedures

Graphite Furnace Atomic Absorption Spectrometry - EPA 600/4-79-020 Methods 204.2, 206.2, 239.2, 270.2, and 279.2; EPA SW-846 Methods 7041, 7060, 7740, and 7841. GEL SOP GL-MA-E-005 for Thermo Jarrell Ash Auto AA 4000 Furnace describes the steps necessary to determine the following trace elements in ultra pure water, drinking water, and groundwater: Antimony, Arsenic, Lead, Selenium, and Thallium. Aqueous samples and extracts are prepared following EPA SW-846 Method 3020 and GEL SOP GL-MA-E-007 for Acid Digestion of Aqueous Samples and Extracts for Analysis by GFAA. Sediment, sludge, and soil are prepared following EPA SW-846 Method 3050A and GEL SOP GL-MA-E-009 for Acid Digestion of Sediments, Sludges, and Soils.

Mercury - EPA 600/14-79-020 Methods 245.2 and 245.5; EPA SW-846 Methods 7470 and 7471. GEL SOP GL-MA-E-001 for Determination of Mercury by Cold Vapor Atomic Absorption Spectrometry details the steps necessary to determine total mercury in surface water, saline water, wastewater, industrial effluent, sanitary sewage, soil, sediment, bottom deposit, sludge, oil, and TCLP extracts using a Bacharach MAS-50 Mercury Analyzer. SOP GL-MA-E-010 for Mercury Analysis Using the Leeman PS200 Automated Mercury Analyzer describes the steps necessary to determine mercury in surface and saline water, wastewater, industrial effluent, sanitary sewage, soil, sediment, bottom deposit, sludge, oil, and TCLP extracts using the Leeman PS200 Automated Mercury Analyzer.

Metals by Inductively Coupled Plasma - EPA 600/4-79-020 Method 200.7; EPA SW-846 Method 6010. GEL SOP GL-MA-E-002 for Determination of Metals by ICP with the Thermo Jarrell Ash ICAP-61E Spectrometer and SOP GL-MA-E-011 for Determination of Metals by ICP with the Thermo Jarrell Ash Enviroll Spectrometer describe the steps necessary to determine the concentration of metals in groundwater, industrial waste, soil, sludge, sediment, TCLP extracts, and other solid and aqueous wastes. For improved detection limits, ultrasonic nebulization is used. The most common analyses include the following elements: Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium,

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Manganese, Molybdenum, Nickel, Phosphorus, Potassium, Selenium, Silicon, Silver, Sodium, Sulfur, Thallium, Tin, Uranium, Vanadium, and Zinc. Other elements are added as required. Surface and groundwater samples are prepared following EPA SW-846 Method 3005 and GEL SOP GL-MA-E-006 for Acid Digestion of Total Recoverable or Dissolved Metals in Surface and Groundwater Samples for Analysis by FLAA or ICP. Aqueous samples and extracts are prepared following EPA SW-846 Method 3010A and GEL SOP GL-MA-E-008 for Acid Digestion of Total Metals in Aqueous Samples and Extracts for Analysis by FLAA or ICP. Sediment, sludge, and soil are prepared following EPA SW-846 Method 3050A and GEL SOP GL-MA-E-009 for Acid Digestion of Sediments, Sludges, and Soils.

Zeeman Graphite Furnace Elemental Analysis - EPA 600/4-79-020 Methods 204.2, 206.2, 239.2, 270.2, and 279.2; EPA SW-846 Methods 7041, 7060, 7421, 7740, and 7841. GEL SOP GL-MA-E-003 for Perkin-Elmer Zeeman 5100 Graphite Furnace Atomic Absorption Spectrometer describes the steps necessary to determine the following elements in drinking water, groundwater, wastewater, soil, and miscellaneous waste: Antimony, Arsenic, Lead, Selenium, and Thallium. Aqueous samples and extracts are prepared following EPA SW-846 Method 3020 and GEL SOP GL-MA-E-007 for Acid Digestion of Aqueous Samples and Extracts for Analysis by GFAA. Sediment, sludge, and soil are prepared following EPA SW-846 Method 3050A and GEL SOP GL-MA-E-009 for Acid Digestion of Sediments, Sludges, and Soils.

# C. General Inorganic Procedures

Acidity - EPA 600/4-79-020 Method 305.1. This is a titrimetric procedure used to determine the acidity present in water and wastewater.

Alkalinity - EPA 600/4-79-020 Method 310.1; SM 17th edition Method 2320-B. GEL SOP GL-GC-E-033 for Alkalinity - Total, Bicarbonate, Carbonate, Hydroxide, and Phenolphthalein describes the steps necessary to titrimetrically determine the total alkalinity in groundwater, drinking water, and domestic and industrial wastewater.

Ammonia Nitrogen, Automated - EPA 600/4-79-020 Method 350.1. GEL SOP GL-GC-E-019 for Ammonia Determination by TRAACS 800 Methodology describes the steps necessary to colorimetrically determine ammonia in groundwater, drinking water, and domestic and industrial wastewater.

Biochemical Oxygen Demand (BOD) - EPA 600/4-79-020 Method 405.1; SM 17th edition Method 5210. GEL SOP GL-GC-E-045 for Biochemical Oxygen Demand describes the steps necessary to determine the biochemical oxygen demand in groundwater, drinking water, and domestic and industrial wastewater.

Carbon Dioxide - SM 17th edition Method 4500-CO₂-D; EPA 600/4-79-020 Method 310.1. GEL SOP GL-GC-E-032 for Carbon Dioxide (Total and Free) By Calculation describes the steps necessary to calculate total and free carbon dioxide in groundwater, wastewater, and drinking water using pH and alkalinity concentrations.

Cation Exchange Capacity - SW-846 Methods 9080 and 9081. GEL SOP GL-GC-E-042 for Cation-Exchange Capacity of Soils (Ammonium Acetate) describes the steps necessary to determine the cation exchange capacity of a soil based on the ammonium content of the treated soil. SOP GL-GC-E-043 for Cation Exchange Capacity of Soils

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(Sodium Acetate) describes the steps necessary to determine the cation exchange capacity of a soil based on the sodium content of the treated soil.

Chemical Oxygen Demand (COD) - EPA 600/4-79-020 Method 410.4. GEL SOP GL-GC-E-061 for Chemical Oxygen Demand (COD) Digestion Reactor Method describes the steps necessary to colorimetrically determine the chemical oxygen demand in groundwater, drinking water, and domestic and industrial wastewater.

Chloride - SM 17th edition Method 4500-Cl-B. GEL SOP GL-GC-E-015 for Chlorides by Silver Nitrate describes the steps necessary to titrimetrically determine chloride in groundwater, drinking water, and domestic and industrial wastewater.

Colliert® - EPA 814B-92-002. GEL SOP GL-GC-E-005 for Colliert® describes the steps necessary for simultaneous detection, specific identification, and confirmation of total coliform and Escherichia coli (E. coli) in drinking water.

Color - EPA 600/4-79-020 Method 110.3. GEL SOP GL-GC-E-036 for Color Determination by Spectrophotometer describes the steps necessary to spectrophotometrically determine color in groundwater, drinking water, and domestic and industrial wastewater.

Conductivity - SM 17th edition Method 2510. GEL SOP GL-GC-E-009 for Conductivity describes the steps necessary to determine specific conductance in groundwater, drinking water, and domestic and industrial wastewater.

Corrosivity by pH - EPA SW-846 Method 9041. GEL SOP GL-GC-E-023 for Corrosivity by pH describes the steps necessary to determine the corrosive characteristic of a sample based on pH for groundwater, drinking water, domestic and industrial wastewater, soil, sludge, and oil.

Corrosivity toward Steel - EPA SW-846 Method 1110. GEL SOP GL-GC-E-029 for Corrosivity Toward Steel describes the steps necessary to determine corrosivity in groundwater, drinking water, and domestic and industrial wastewater.

Cyanide Amenable to Chlorination - EPA 600/4-79-020 Methods 335.1 and 335.3: EPA SW-846 Method 9012. GEL SOP GL-GC-E-040 for Cyanide Amenable to Chlorination by Alpkem Flow Solution RFA describes the steps necessary to determine cyanide amenable to chlorination in groundwater, drinking water, and domestic and industrial wastewater.

Cyanide by Rapid Flow Analyzer - EPA 600/4-79-020 Method 335.3; EPA SW-846 Methods 9010A and 9012A. GEL SOP GL-GC-E-014 for Total Cyanide by Alpkem Flow Solution RFA describes the steps necessary for automatic colorimetric measurement of cyanide in groundwater, drinking water, domestic and industrial wastewater, soil, sludge, and oil.

Dissolved Oxygen - SM 17th edition Method 4500-O-G. GEL SOP GL-GC-E-059 for Dissolved Oxygen Analysis by Membrane Electrode Method describes the steps necessary to determine the dissolved oxygen content in groundwater, drinking water, and domestic and industrial wastewater.

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Extractable Organic Halogen (EOX) - Dohrmann DX-20 A/B Operations Manual. GEL SOP GL-GC-E-060 for Extractable Organic Halides describes the steps necessary to determine extractable organic halides in soil, solid, and oil.

Fecal Coliform by Membrane Filter - SM 17th edition Method 9222-D. GEL SOP GL-GC-E-031 for Fecal Coliform by Membrane Filter describes the steps necessary to determine fecal coliform by membrane filter in groundwater, drinking water, and domestic and industrial wastewater.

Fecal Coliform Most Probable Number (MPN) - SM 17th edition Method 9221-C. GEL SOP GL-GC-E-034 for Fecal Coliform Most Probable Number (5 Tube Dilution) describes the steps necessary to determine fecal coliform by multiple tubes in groundwater, drinking water, domestic and industrial wastewater, soil, and sludge.

Flammability Screen - ASTM D4982-89. GEL SOP GL-GC-E-030 for Flammability Potential Screening Analysis of Waste describes the steps necessary to indicate the fire producing potential of domestic and industrial wastewater, soil, sludge, and oil.

Flashpoint - EPA SW-846 Method 1010. GEL SOP GL-GC-E-027 for Pensky-Martens Closed Cup Flashpoint describes the steps necessary to determine the flashpoint in domestic and industrial wastewater and oil.

Fluoride - EPA-600/4-79-020 Method 340.2. GEL SOP GL-GC-E-002 for Fluoride describes the steps necessary to determine fluoride by ion selective electrode in groundwater, drinking water, domestic and industrial wastewater, soil, sludge, and oil.

Formaldehyde - AOAC Methods 20.062 and 20.063. GEL SOP GL-GC-E-049 for Formaldehyde describes the steps necessary to spectrophotometrically determine formaldehyde in groundwater, drinking water, domestic and industrial wastewater, soil, and sludge.

Hardness - SM 17th edition Method 2340-B. GEL SOP GL-GC-E-025 for Hardness by Calculation describes the steps necessary to calculate hardness using calcium and magnesium concentrations in groundwater, drinking water, wastewater, soil, and sludge.

Heating Value (BTU) - ASTM Methods D 240-87, E 711-87, and 2382-88. GEL SOP GL-GC-E-048 for Heating Value Determination by Bomb Calorimeter describes the steps necessary to determine the heat of combustion in soil, sludge, oil, and miscellaneous liquid and solid samples.

Hexavalent Chromium - EPA SW-846 Method 7196A. GEL SOP GL-GC-E-044 for Colorimetric Determination of Chromium, Hexavalent describes the steps necessary to colorimetrically determine hexavalent chromium in groundwater, drinking water, and domestic and industrial wastewater.

Hydrazine - ASTM, Part 31, D1385-78/1981. This is a colorimetric procedure for the determination of hydrazine in water and wastewater.

Inhibitory Residue - SM 17th edition, Method 9020-B, Part 3. GEL SOP GL-GC-E-038 for Inhibitory Residue Analysis describes the steps necessary to determine if inhibitory effects are present in a detergent.

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Inorganic Anions by Ion Chromatography - SM 17th edition Method 4110-B; EPA 600/4-84-017 Method 300.0. GEL SOP GL-OA-E-025 for Ion Chromatography describes the steps necessary to determine the following compounds by ion chromatography in groundwater, drinking water, domestic and industrial wastewater, soil, sludge, and oil: Bromide, Chloride, Fluoride, Nitrate, Nitrite, Sulfate, and Oxalate.

Iodine - SM 16th edition, Method 415A. GEL SOP GL-GC-E-003 for Iodine describes the steps necessary to spectrophotometrically determine iodine in groundwater, drinking water, and domestic and industrial wastewater.

Laboratory Water Toxicity - SCDHEC; SM 17th edition Methods 9215A and 9020B. GEL SOP GL-GC-E-039 for Laboratory Water Toxicity Analysis describes the steps necessary to determine if toxicity effects are present in laboratory water.

Nitrate/Nitrite - EPA-600/4-79-020 Method 353.1. GEL SOP GL-GC-E-041 for Nitrate/Nitrite by TRAACS 800 Methodology describes the steps necessary to colorimetrically determine nitrate/nitrite in groundwater, drinking water, and domestic and industrial wastewater.

Odor - EPA 600/4-79-020 Method 140.1. GEL SOP GL-GC-E-050 describes the steps necessary to determine the threshold odor of drinking water, surface water, saline water. and domestic and industrial wastewater.

Oil and Grease - EPA 600/4-79-020 Method 413.1; EPA SW-846 Method 9070. GEL SOP GL-GC-E-020 for Oil and Grease describes the steps necessary to gravimetrically determine oil and grease in groundwater, drinking water, and domestic and industrial wastewater.

Orthophosphate - EPA 600/4-79-020 Method 365.2. GEL SOP GL-GC-E-046 for Orthophosphate describes the steps necessary to colorimetrically determine orthophosphate in groundwater, drinking water, domestic and industrial wastewater, soil, and sludge.

Paint Filter Test - EPA SW-846 Method 9095. GEL SOP GL-GC-E-010 for Paint Filter Test describes the steps necessary to determine the presence of free liquid in soil and sludge.

Percent Water by Karl Fischer Titration - ASTM Method E-203. This is a titrimetric procedure used to determine the presence of water in various matrices. Interferences with this procedure are free alkali, oxidizing and reducing agents. mercaptans, certain basic nitrogenous substances, and other materials that react with iodine.

pH - EPA 600/4-79-020 Method 150.1; EPA SW-846 Methods 9041 and 9045. GEL SOP GL-GC-E-008 for pH describes the steps necessary to electrometrically determine pH for groundwater, drinking water, domestic and industrial wastewater, soil, sludge, and oil.

Phenol by Rapid Flow Analyzer - EPA 600/4-79-020 Method 420.2. GEL SOP GL-GC-E-013 for Total Phenol by Alpkem Flow Solution RFA describes the steps necessary for automatic colorimetric measurement of phenol in groundwater, drinking water, and domestic and industrial wastewater.

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Release of Sulfide - Hach Model HS-C Hydrogen Sulfide Test Kit. GEL SOP GL-GC-E-016 for Release of Cyanide and Sulfide describes the steps necessary to determine if a waste classifies as reactive due to the release of sulfide.

Release of Cyanide - Method developed at GEL. GEL SOP GL-GC-E-016 for Release of Cyanide and Sulfide describes the steps necessary to determine, using a cyanide test strip, if a waste classifies as reactive due to the release of cyanide.

Residual Chlorine - EPA 600/4-79-020 Method 330.5. This is a colorimetric procedure used for the determination of total residual chlorine in water and wastewater.

Salinity - SM 16th edition Method 210-C. GEL SOP GL-GC-E-051 for Argentometric describes the steps necessary to determine salinity in water and wastewater.

Specific Gravity - SM 16th edition Method 213-E. Specific gravity is determined by comparing the weight of a volume of mud, sludge, or industrial waste with the weight of an equal volume of distilled water.

Standard Plate Count - SM 17th edition Method 9215. GEL SOP GL-GC-E-053 for Heterotrophic Plate Count (Standard Plate Count) describes the steps necessary to perform a standard plate count on groundwater and drinking water.

Sulfide - EPA 600/4-79-020 Method 376.2. GEL SOP GL-GC-E-052 for Sulfide (Methylene Blue Method) describes the steps necessary to colorimetrically determine sulfide in groundwater, drinking water, and domestic and industrial wastewater.

Sulfite - SM 17th edition Method 4500-SO₃2--B. GEL SOP GL-GC-E-056 for Sulfite (SO₃²) by Iodometric Method describes the steps necessary to titrimetrically determine sulfite in groundwater, drinking water, domestic and industrial wastewater, and soil.

Surfactants (Methylene Blue Active Substances). EPA 600/4-79-020 Method 425.1 - GEL SOP GL-GC-E-047 for Methylene Blue Active Substance describes the steps necessary to colorimetrically determine surfactants in groundwater, drinking water, domestic and industrial wastewater, and soil.

Total Carbon in Solid Samples (TC) - EPA SW-846 Method 9060. GEL SOP GL-GC-E-062 for Total Carbon and Total Organic Carbon Analysis using the Dohrmann DC-190 Boat Sampler describes the steps necessary to determine total carbon in soil and sludge.

Total Carbon in Water (TC) - EPA 600/4-79-020 Method 415.1; EPA SW-846 Method 9060. GEL SOP GL-GC-E-006 for Total, Total Inorganic, and Total Organic Carbon (TOC) using the Dohrmann DC-80 TOC Analyzer describes the steps necessary to determine total carbon in groundwater, drinking water, and domestic and industrial wastewater.

Total Coliform - SM 17th edition Method 9222-B. GEL SOP GL-GC-E-054 for Total Coliform By Membrane Filter describes the steps necessary to determine the total coliform in groundwater, drinking water, and domestic and industrial wastewater by filtering through a sterile membrane filter.

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Total Coliform Most Probable Number - SM 17th edition Method 9221-B. GEL SOP GL-GC-E-063 for Total Coliform Most Probable Number (5 Tube Dilution) describes the steps necessary to determine total coliform by multiple tubes in groundwater, drinking water, domestic and industrial wastewater, soil, and sludge.

Total Dissolved Solids (TDS) - EPA 600/4-79-020 Method 160.1. GEL SOP GL-GC-E-001 for Total Dissolved Solids describes the steps necessary to gravimetrically determine total dissolved solids in groundwater, drinking water, and domestic and industrial wastewater.

Total Inorganic Carbon in Water (TIC) - EPA 600/4-79-020 Method 415.1; EPA SW-846 Method 9060. GEL SOP GL-GC-E-006 for Total, Total Inorganic, and Total Organic Carbon (TOC) using the Dohrmann DC-80 TOC Analyzer describes the steps necessary to determine total inorganic carbon in groundwater, drinking water, and domestic and industrial wastewater.

Total Kieldahl Nitrogen - EPA-600/4-79-020 Method 351.2. GEL SOP GL-GC-E-055 for Total Kjeldahl Nitrogen, Total Organic Nitrogen, and Total Nitrogen by TRAACS 800 Methodology describes the steps necessary to colorimetrically determine total kieldahl nitrogen in groundwater, drinking water, and domestic and industrial wastewater.

Total Nitrogen - EPA 600/4-79-020. GEL SOP GL-GC-E-055 for Total Kieldahl Nitrogen, Total Organic Nitrogen, and Total Nitrogen by TRAACS 800 Methodology describes the steps necessary to calculate total nitrogen in groundwater, drinking water, and domestic and industrial wastewater.

Total Organic Carbon in Solid Samples (TOC) - EPA SW-846 Method 9060. GEL SOP GL-GC-E-062 for Total Carbon and Total Organic Carbon Analysis using the Dohrmann DC-190 Boat Sampler describes the steps necessary to determine total organic carbon in soil and sludge.

Total Organic Carbon in Water (TOC) - EPA 600/4-79-020 Method 415.1; EPA SW-846 Method 9060. GEL SOP GL-GC-E-006 for Total, Total Inorganic, and Total Organic Carbon (TOC) Using the Dohrmann DC-80 TOC Analyzer describes the steps necessary to determine total organic carbon in groundwater, drinking water, and domestic and industrial wastewater.

Total Organic Halogen (TOX) - EPA SW-846 Method 9020. GEL SOP GL-GC-E-007 for Total Organic Halogens using the COSA TOX-10 Analyzer describes the steps necessary to determine total organic halogen in groundwater and drinking water.

Total Organic Nitrogen - EPA 600/4-79-020. GEL SOP GL-GC-E-055 for Total Kjeldahl Nitrogen, Total Organic Nitrogen, and Total Nitrogen by TRAACS 800 Methodology describes the steps necessary to calculate total organic nitrogen in groundwater, drinking water, and domestic and industrial wastewater.

Total Petroleum Hydrocarbons, Gravimetric - SM 17th edition Methods 5520E and 5520F; EPA SW-846 Methods 9070A and 9071. GEL SOP GL-GC-E-017 for Gravimetric Total Petroleum Hydrocarbons (TPH) Liquids describes the steps necessary to gravimetrically determine total petroleum hydrocarbons in groundwater, drinking water. and domestic and industrial wastewater. GEL SOP GL-GC-E-018 for Oil and Grease

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Gravimetric For Solids describes the steps necessary to gravimetrically determine total petroleum hydrocarbons in soil and sludge.

Total Petroleum Hydrocarbons, Spectrophotometric - EPA 600/4-79-020 Method 418.1. GEL SOP GL-GC-E-022 for Determination of Total Petroleum Hydrocarbons Liquid by Infrared Spectrophotometry describes the steps necessary to spectrophotometrically determine total petroleum hydrocarbons in groundwater, drinking water, and domestic and industrial wastewater. GEL SOP GL-GC-E-021 for Determination of Total Petroleum Hydrocarbons Solid by Infrared Spectrophotometry describes the steps necessary to spectrophotometrically determine total petroleum hydrocarbons in soil and sludge.

Total Phosphorus - EPA 600/4-79-020 Method 365.1. GEL SOP GL-GC-E-026 for Total Phosphorus describes the steps necessary to colorimetrically determine total phosphorus in groundwater, drinking water, and domestic and industrial wastewater.

Total Solids (TS) - EPA 600/4-79-020 Method 160.3. GEL SOP GL-GC-E-011 for Total Solids describes the steps necessary to gravimetrically determine total solids in groundwater, drinking water, domestic and industrial wastewater, soil, and sludge.

Total Suspended Solids (TSS) - EPA-600/4-79-020 Method 160.2. GEL SOP GL-GC-E-012 for Total Suspended Solids describes the steps necessary to gravimetrically determine total suspended solids in groundwater, drinking water, and domestic and industrial wastewater.

Turbidity - EPA 600/4-79-020 Method 180.1. GEL SOP GL-GC-E-037 for Turbidity describes the steps necessary to determine turbidity in groundwater, drinking water, and domestic and industrial wastewater.

Volatile Solids (Ash) - SM 17th edition Methods 2540-E and 2540-G. GEL SOP GL-GC-E-057 for Volatile Solids and % Ash-550 - Procedure for Water Samples describes the steps necessary to gravimetrically determine the volatile solids in groundwater, drinking water, and domestic and industrial wastewater. GEL SOP GL-GC-E-058 for Volatile Solids and % Ash-550 - Procedure for Solid and Semisolid Samples describes the steps necessary to gravimetrically determine the volatile solids in soil and sludge.

Volatile Suspended Solids (VSS) - EPA 600/4-79-020 Methods 160.2 and 160.4; SM 17th edition 2540-E. GEL SOP GL-GC-E-035 for Volatile Suspended Solids describes the steps necessary to gravimetrically determine volatile suspended solids in groundwater, drinking water, and domestic and industrial wastewater.

# D. Radiochemistry Procedures

Carbon-14 - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-003 for Carbon-14 in Water describes the steps necessary to conduct the analysis for Carbon-14 in water using a liquid scintillation counter. SOP A-003B for The Determination of Carbon-14 in Soil describes the steps necessary to conduct the analysis for Carbon-14 in soil using a liquid scintillation counting system.

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Gamma Isotopes - EPA Method 901.1. EPI SOPs A-013 for Gamma Isotopes in Water and A-013B for Determination of Gamma Isotopes in Soil describe the steps necessary to determine gamma isotopes in water and soil respectively using a gamma spectrometer.

Gross Alpha and Beta - EPA Method 900.0. EPI SOPs A-001 for Determination of Gross Alpha and Gross Non-Volatile Beta in Water and A-001B for Determination of Gross Alpha and Gross Non-Volatile Beta in Soil describe the steps necessary to determine gross alpha and non-volatile beta in water and soil respectively using a gas-flow proportional counter.

Iodine-129 - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-006 for The Determination of Iodine-129 in Water describes the steps necessary to determine I-129 in water using an x-ray spectrometer. EPI SOP A-006B for The Determination of I-129 in Soil describes the steps necessary to determine I-129 in soil using a micro-Vax 3100 with x-ray or extended range detectors.

Iodine-131 - EPA Method 902. EPI SOP A-017 for The Determination of Iodine-131 in Water describes the steps necessary to conduct the analysis for I-131 in water using a gasflow proportional counter.

Isotopic Neptunium, Plutonium, and Thorium - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-012 for Determination of Thorium, Plutonium, and Neptunium in Water describes the steps necessary to conduct the analysis for isotopic plutonium, thorium, and neptunium in water using a gas-flow proportional counter, a liquid scintillation counter, and an alpha spectrometer. SOP A-012B for Isotopic Neptunium and Plutonium in Soil and Vegetation describes the steps necessary to conduct the analysis for alpha emitting plutonium and neptunium isotopes in soil and vegetation using the micro-Vax 3100 and gas-flow proportional counter. If analyzing for Pu-241, the liquid scintillation counter is used. EPI SOP A-012C for The Determination of Isotopic Thorium in Soil describes the steps necessary to conduct the analysis for isotopic thorium in soil using the micro-Vax 3100 and alpha spectrometers. EPI SOP A-012D for Isotopic Determination of Thorium, Plutonium, and Neptunium in Air Filters describes the steps necessary to conduct the analysis of isotopic plutonium, thorium, and neptunium in air filters using a gas-flow proportional counter and alpha spectrometers.

Isotopic Uranium - EPA; EML; LAHEC, 10300-M, Vol. 1. EPI SOP A-011C for Determination of Isotopic Uranium in Air Filters describes the steps necessary to determine uranium in air filters using the micro-Vax 3100 and alpha spectrometers.

Isotopic Uranium, Americium, and Curium - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-011 for Isotopic Determination of Uranium, Americium, and Curium in Water describes the steps necessary to determine uranium, americium, and curium in water using an alpha spectrometer. EPI SOP A-011B for The Determination of Uranium, Americium, and Curium in Soil and Vegetation describes the steps necessary to determine uranium, americium, and curium in soil and vegetation using the micro-Vax 3100 and alpha spectrometers.

Lead-210 - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-018 for The Determination of Lead-210 in Water describes the steps necessary to conduct the analysis for Lead-210 in water using the gross alpha beta proportional counting system.

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Nickel-59 and Nickel-63 - EML "Determination of Ni-59 and Ni-63 in Water and Soil". EPI SOP A-022 for The Determination of Ni-59 and Ni-63 in Soil and Water describes the steps necessary to conduct the analysis of nickel-59 and/or nickel-63 in soil and water using a low energy germanium detector and a liquid scintillation counter.

Phosphorus-32 - EML. EPI SOP A-019 for The Determination of Phosphorus-32 in Soil and Water describes the steps necessary to conduct the analysis for Phosphorus-32 in soil and water using the liquid scintillation counter.

Polonium - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-016 for The Determination of Polonium in Water describes the steps necessary to conduct the analysis for isotopic alpha emitting polonium in water using the micro-Vax 3100 and alpha spectrometers. EPI SOP A-016B for The Determination of Polonium in Soil describes the steps necessary to conduct the analysis for isotopic alpha emitting polonium in soil using the Micro-Vax 3100 and alpha spectrometers.

Promethium-147 - Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-020 for The Determination of Promethium-147 in Soil and Water describes the steps necessary to conduct the analysis for promethium-147 in soil and water using the liquid scintillation counter.

Radionuclides - 1993 Annual Book of ASTM Standards. EPI SOP A-021 for Soil Sample Preparation for the Determination of Radionuclides describes the steps necessary to prepare soil samples for radionuclide determination using a Ball Mill and an analytical balance.

Radium-223 and Radium-224 - DOE RESL A-3-1. EPI SOP A-024 for the Determination of Radium-223 and Radium-224 Isotopes in Water describes the steps necessary to conduct the analysis for Radium-223 and 224 in water using the alpha spectrometer and gamma spectrometer.

Radium-226 - EPA Method 903.1. EPI SOP A-008 for Radium-226 in Water describes the steps necessary to determine radium-226 in water using a lucas cell counter.

Radium-228 - EPA Method 904; Method developed by Dr. Burnett in conjunction with FSU. EPI SOP A-009 for The Determination of Radium-228 in Water describes the steps necessary to determine radium-228 in water using the Gross Alpha Beta Proportional Counting System, the gamma spectrometer, and associated electronics and data reduction packages.

Radon-222 - EPI SOPs A-007 for Determination of Radon-222 in Water and A-007B for Radon-222 in Soil describe the steps necessary to determine radon-222 in water and soil respectively using a liquid scintillation counter.

Strontium 89/90 - EPA Method 905. EPI SOP A-004 for The Determination of Strontium 89/90 in Water and Soil describes the steps necessary to determine isotopic Sr-89 and Sr-90 in water and soil using a gas-flow proportional counter.

Technetium-99 - Method supplied by the DOE. EPI SOP's A-005 for Determination of Technetium-99 in Water and A-005B for Determination of Technetium-99 in Soil describe the steps necessary to determine technetium-99 in water and soil respectively using a liquid scintillation counter.

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Total Alpha Radium - EPA Method 903. EPI SOP's A-010 for Total Alpha Radium Isotopes in Water and A-010B for Total Alpha Radium Isotopes in Soil describe the steps necessary to determine total alpha radium in water and soil respectively using a gas-flow proportional counter.

Total Uranium - EPI SOP A-023 for Total Uranium in Water and Soils by Laser Phosphorescence describes the steps necessary to conduct the analysis for total uranium in water and soil using the Chemchek Kinetic Laser Phosphorimeter System.

Tritium - EPA Method 906.0. EPI SOPs A-002 for Determination of Tritium in Water; A-002B for Determination of Tritium in Soil; A-002C Determination of Tritium in Vegetation; A-002D for Determination of Tritium in Air Filters; and A-002E for Determination of Tritium in Water by Distillation describe the steps necessary to determine tritium in water, soil, vegetation, air filters, and water by distillation respectively using a liquid scintillation counter.

## E. Air Analysis Procedures

Base/Neutral and Acid Extractable Compounds - 40 CFR Part VIII Method 625; EPA SW-846 Method 8270. GEL SOP GL-OA-E-009 for Semivolatile Analysis by Gas Chromatograph/Mass Spectrometer describes the steps necessary to determine semivolatile organic compounds by gas chromatography/mass spectrometry in ambient air using a mass selective detector. Samples are prepared according to SOP GL-OA-E-024 for Cleanup and Extraction of Polyurethane Foam Cartridge (PUF) For Semivolatile Analysis.

Inorganic Anions by Ion Chromatography - SM 17th edition Method 4110-B; EPA 600/4-84-017 Method 300.0. GEL SOP GL-OA-E-025 for Ion Chromatography describes the steps necessary to determine the following compounds by ion chromatography in air and stack samples: Bromide, Chloride, Fluoride, Nitrate, Nitrite, Sulfate, and Oxalate.

Volatile Compounds - EPA TO-14 Compendium. GEL SOP GL-OA-E-028 for the Determination of Volatile Organic Compounds (VOC's) in Ambient Air using Summa Passivated Canister Sampling and Gas Chromatograph/Mass Spectrometer analysis describes the steps necessary in the determination of VOC's in ambient air, indoor air, landfill gases, and other gaseous samples.

## VIII. DATA REDUCTION, REVIEW, VALIDATION, AND REPORTING

Analytical data is generated from the various instruments and analytical procedures at GEL. The data is taken from its raw form and translated to a form that can be used by the client. The interpretation of raw data and the calculations associated with transforming raw data into usable data is data reduction.

Using measurement instrumentation with integrated data systems is one of the best ways to avoid mathematical errors that can occur during the manual manipulation of numbers in the calculations of results. The automatic data systems and the LIMS used by GEL are described in Section V. Figure 4 shows the flow of the sample through the laboratory, including the data review process after the data reduction.

## A. Data Reduction

Instrument software calculates parameter concentration for most of the analyses performed at GEL. For analyses where instrument software is not available, the following calculations are used:

<u>Colorimetric Methods</u>: Results are calculated by comparing sample absorbance or transmittance measurements with a calibration curve of standard peak absorbance or transmittance versus concentration:

(mg/L from standard curve) x  $D_f$  = Concentration in mg/L

Where:

 $D_t = Dilution Factor$ 

Titration Methods: The calculation is:

$$C = \frac{(A - B)}{S} \times N$$

Where:

C = Concentration in mg/L

A = ml of titrant used for sample

B = ml of titrant used for blank

N = Normality of titrant

S = Volume of sample in ml

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Gravimetric Methods: The calculation is:

$$C_1 = \frac{Fw - Iw}{Ss}$$

Where:

 $C_1 = \text{Concentration in mg/kg or mg/L}$ 

 $F_w = Final weight in grams$ 

 $I_w = Initial$  weight in grams

 $S_s$  = Sample size in grams or liters

Then:  $C_F = C_1 - C_B$ 

Where:

C_F = Final concentration in mg/kg or mg/L

 $C_1$  = Concentration of sample in mg/kg or mg/L

C_B = Concentration of blank in mg/kg or mg/L

The concentration generated by the instrument is entered into LIMS along with the preparation and dilution factors. The LIMS calculates the reported value for each parameter. The concentration manually calculated is usually entered into LIMS as the reported value. The individual analytical SOPs contain specific details on the information required for the LIMS for each analysis. The SOPs and instrument manuals provide detailed descriptions of the calculations performed manually and electronically.

#### B. Data Review

The data review process is broken down into specific levels. Three levels of review are performed by the laboratory. An additional level of review is conducted by the Analytical Report Specialist. The review process is explained in GEL SOP GL-LB-E-005 for Data Review/Validation.

When analysis is complete, the analyst conducting the procedure reviews 100 percent of the raw data and QC data for each test. Once the data has been entered into the LIMS system, a data package is prepared. The package includes standardizations, calculations, raw data, quality control criteria as established by methodologies, and a computer generated "Batch Report". If any criteria were not met, the data is considered out of control and actions to correct the nonconforming data are taken before the data can be sent to the next level of review.

The second level of data review is a technical review performed by a peer. This is a review of the completed data package including review of the standardizations, calculations, raw data, quality control criteria as established by methodologies, and a check for transcription errors. If any criteria were not met, the data is considered out of control and actions to correct the nonconforming data are taken before the data is sent to the final

level of review. Any nonconforming items which have been accepted are noted so that specific flags are reported with the data when the Certificate of Analysis is printed.

The third level of data review is conducted by the Group Leader or Section Head. This is a technical review of the completed reviewed data package. If any criteria were not met, the data is considered out of control and actions to correct the nonconforming data are taken before the data is sent to the final level of review. Any nonconforming items which have been accepted are noted so that specific flags are reported with the data when the Certificate of Analysis is printed.

When all required parameters for a specific sample have been completed by the laboratory, a sample report is generated and reviewed by an Analytical Report Specialist. This report is reviewed for completeness, special comments, and flags as explained in GEL SOP GL-QS-E-009 for Sample Report Preparation and Review.

#### С. Data Validation

Data validation is a process by which data generated by the laboratory is reviewed for completeness, correctness, and acceptability as it relates to specific project and method requirements. The validation process addresses the required technical and QC analyses, the frequency of these analyses, and acceptability of the analyses based on criteria in the project plan. The documentation review of preparation logs, standard logs, calibration logs, instrument logs, data interpretation, checklists, custody documents, data reports, and certificates of analysis are conducted on a sample and project specific basis. A data validation report is written after completion of this review and presented to Senior Management

#### D. Reporting Format

Data entered into the LIMS system is used to generate a client's Certificate of Analysis (COA). The versatility of LIMS allows for specialized reporting formats based upon a client's needs. All COA's contain the client's sample identification, date sample was collected, date sample was received by the laboratory, and sample due date.

Routine COA's also contain the parameter, method identification, analyst's initials, and analysis date. Results are reported as a single value in concentrations at or above a samples practical quantitation limit. Those parameters found to be below or undetected are reported as less than (<) the sample's practical quantitation limit. An example of a Routine Certificate of Analysis is shown in Appendix 7.

A Level 2 COA contains all the information found in a routine COA. In addition, the analyte concentration in the sample and the analyte's Practical Quantitation Limit (POL) are reported. An example of a Level 2 COA is shown in Appendix 8.

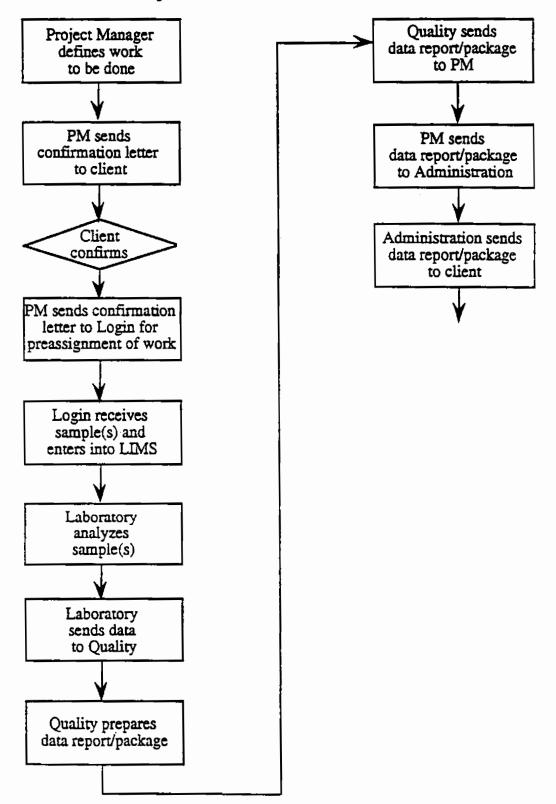
A Level 3 COA contains all the information found in a Level 2 COA. In addition, this report includes results for the method blank, matrix spike recovery, matrix spike duplicate, and sample duplicate data. An example of a Level 3 COA is shown in Appendix General Engineering Laboratories, Inc. GEL QAP No.: GL-QS-B-001 - Rev. 9 Quality Assurance Plan

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Electronic Deliverable Data (EDD) are generated according to a client's contractual needs. These data reports are reviewed in the same manner as hardcopy COA's. The process of review for these formats is given in GEL SOP GL-CS-E-002 for Internal Review of Contractually Required Quality Criteria for Client Package Delivery.

The versatility of the LIMS allows for specialized non-routine reporting formats to be generated at a client's request.

FIGURE 4
General Engineering Laboratories, Inc.
Analytical Services Flow Chart



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## IX. LABORATORY QUALITY CONTROL CHECKS

## A. Overview

The goals of GEL Quality Control (QC) system are to:

1. Define the quality of data produced by monitoring the precision and accuracy of the analytical results.

2. Improve the overall quality of data.

Monitor technical compliance with contractual QC requirements.

Evaluate laboratory performance.

5. Upgrade the overall quality of laboratory performance.

In order to meet these goals, GEL implements the quality control requirements described in the reference methods that it uses. Specific QC requirements are described in the appropriate SOP under consideration.

It is GELs' policy to ensure that all applicable QC requirements are met for each analysis reported to clients.

When analyses are found to be out of control, the actions described in the appropriate analytical SOP are followed.

#### Statistical Process Control Database

A Process Control System is a feedback system that uses process data information to assess status, diagnose/decide potential risks, and take appropriate actions. GEL has a system that takes data as it is generated by the analyst and stores it in the LIMS. The data is used to generate process control limits for the specific tests or processes. The Group Leader is responsible for periodically reviewing this data to check for inconsistencies and to generate the quarterly control limits. The documented limits are stored in Quality Records. A copy of the control limits are returned to the Group Leader for use in his/her area.

### Metals Analyses Quality Control Checks

The EPA SW-846 Methodologies for the Analysis of Solid and Hazardous Waste is the standard reference analytical procedures for GEL. These documents outline the criteria for the QC actions required for metals analyses in environmental samples. The following quality control checks are analyzed by our laboratory for metals:

- 1. Initial Calibration Verification
- 2. Continuing Calibration Verification
- 3. Initial Calibration Blank
- 4. Continuing Calibration Blank
- 5. Method Blank
- 6. Interference Check Sample, where applicable
- 7. Matrix Spike/Matrix Spike Duplicate
- 8. Analytical Spike/Serial Dilution, where appropriate
- 9. Sample Duplicate
- Laboratory Control Sample

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- 11. Method of Standard Addition, where applicable
- 12. CRDL Check Standard

# General Inorganic Analyses Quality Control Checks

Standard Methods for Analysis of Water and Wastewater and EPA SW-846 outline the criteria for the quality control required for general inorganic analyses in environmental samples. The following quality control checks are analyzed by our laboratory for general inorganic chemistry parameters:

- 1. Initial Calibration Verification
- Continuing Calibration Verification
   Initial Calibration Blank, where appropriate
- 4. Continuing Calibration Blank, where appropriate
- 5. Method Blank
- 6. Matrix Spike/Matrix Spike Duplicate
- Sample Duplicate
   Laboratory Control Sample

## Organic Analyses Quality Control Checks

The criteria for the quality control required for analysis of environmental samples for organic parameters are outlined in EPA SW-846 and the EPA 600 Series. The following quality control checks are analyzed by the laboratory for organic chemistry parameters:

- Surrogate spike
- Blank spike
- 3. Matrix spike/Matrix spike duplicate
- 4. Method blank
- 5. Sample duplicate6. GC/MS tuning and mass calibration
- 7. Initial calibration
- 8. Continuing calibration verification

## Radiochemistry Analyses Ouality Control Checks

The EPA 900 Series Protocols for Measurement of Radioactivity in Drinking Water and EML Procedures Manual, No. HASL-300 outline the criteria for the quality control required for radiochemistry analyses in environmental samples. The following quality control checks are analyzed by our laboratory for radiochemistry parameters:

- 1. Initial calibration
- 2. Method blank
- Matrix spike
- Sample duplicate
   Laboratory control sample

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#### В. Quality Control for Metals Analysis

## Instrument Performance OC for Analysis of Metals

Inductively Coupled Plasma (ICP)

<u>Initial Calibration</u> - The objective in establishing compliance requirements for satisfactory instrument calibration is to ensure that the instrument is capable of producing acceptable data. The analytical methods specify that instruments must be calibrated each time the instrument is used on a daily basis. The number of calibration standards is specified in each analytical SOP. Calibration standards must be prepared using the same type of acid or combination of acids as the digestion procedure and at the expected concentration of the samples. In addition, GEL analyzes a standard at or below the reported level of detection.

Initial Calibration Verification - Initial calibration verification (ICV) is performed immediately following instrument calibration by analyzing an independent solution to verify and document the accuracy of the initial calibration for each analyte. When ICV results exceed the specified control limits, the analysis must be terminated, the problem corrected. the instrument recalibrated, and the calibration reverified.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) - An initial calibration blank (ICB) is analyzed each time the instrument is calibrated. A continuing calibration blank (CCB) is analyzed at the end of the run and at a frequency of 10% during the run. The ICB and CCB serve as a monitor of instrument drift and possible contamination. If the ICB or CCB result is greater than the reporting limit, the analysis is terminated, the problem identified and corrected, and the instrument recalibrated.

Continuing Calibration Verification (CCV) - To assure calibration accuracy and monitor instrument performance during each analysis, a continuing calibration verification (CCV) standard is analyzed at a frequency of 10% or every 2 hours, whichever is more frequent. The analyte concentration must be at or near the mid-range level of the calibration. When the CCV results exceed the specified control limits, the analysis is terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

Interference Check Sample - The interference check sample (ICS) is analyzed to verify the ICP's interelement and background correction factors. An ICS must be analyzed at the beginning and end of each analytical run or at a minimum of twice per 8-hour working shift, whichever is more frequent. If the results of the ICS do not meet the specified control limits, the analysis is terminated, the problem corrected, the instrument recalibrated, the calibration reverified, and the samples reanalyzed.

Linear Range Analysis - To verify linearity near the required detection limit, the laboratory must analyze a standard at the concentration level of practical quantitation limit (PQL) or reporting limit. This standard must be run at the beginning and end of each analytical run or at a minimum of twice per 8-hour working shift, whichever is more frequent. The upper limit of the linear range must be verified on an annual basis. The analytically determined concentration of the standard used to define this upper limit must fall within ±5% of the true value. This concentration defines the limit beyond which results cannot be reported without dilution of the sample into the working range.

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# Graphite Furnace Atomic Absorption (GFAA)

<u>Initial Calibration</u> - The objective in establishing compliance requirements for satisfactory instrument calibration is to ensure that the instrument is capable of producing acceptable data. The analytical methods specify that instruments must be calibrated each time the instrument is used on a daily basis. The number of calibration standards is specified in each analytical SOP. Calibration standards must be prepared using the same type of acid or combination of acids as the digestion procedure and at the expected concentration of the samples. In addition, GEL analyzes a standard at or below the reported level of detection.

Initial Calibration Verification - Initial calibration verification (ICV) is performed immediately following instrument calibration by analyzing an independent solution to verify and document the accuracy of the initial calibration for each analyte. When ICV results exceed the specified control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) - An initial calibration blank (ICB) is analyzed each time the instrument is calibrated. A continuing calibration blank (CCB) is analyzed at the end of the run and at a frequency of 10% during the run. The ICB and CCB serve as a monitor of instrument drift and possible contamination. If the ICB or CCB result is greater than the reporting limit, the analysis is terminated, the problem identified and corrected, and the instrument recalibrated.

Continuing Calibration Verification (CCV) - To assure calibration accuracy and monitor instrument performance during each analysis, a continuing calibration verification (CCV) standard is analyzed at a frequency of 10% or every 2 hours, whichever is more frequent. The analyte concentration must be at or near the mid-range level of the calibration. When the CCV results exceed the specified control limits, the analysis is terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

## Method Performance OC for Analysis of Metals

Method Blank (MB) - The analysis of a method blank provides the laboratory and the regulatory agencies a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. At least one method blank of similar matrix as the samples must be prepared and analyzed with each analytical batch of samples. If the concentration of the method blank is above the reporting limit, then any samples with less than 10 times the concentration level identified in the blank must be redigested and reanalyzed. The reported sample results are not corrected for the blank results.

Serial Dilution - Serial dilution analysis is performed to ascertain whether significant physical or chemical interferences exist due to the sample matrix. One sample from each group of samples of a similar matrix type must undergo at least one serial dilution analysis. If the analyte concentration is minimally a factor of 10 above the IDL after dilution, the analysis must agree within 10% of the original determination. For ICP analyses, if the result is not within 10%, a chemical or physical interference effect should be suspected and the data flagged. For GFAA analyses, if the serial dilution or analytical spike fail, a MSA is analyzed.

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Laboratory Control Sample (LCS) - The laboratory control sample (LCS) is designed to serve as a monitor of the efficiency of the analytical process. The LCS must be analyzed on a digestion batch basis. Results of the LCS analyses must fall within established control limits. Results which fall outside the specified control limits are indicative of an analytical problem related to the digestion/sample preparation procedures and/or instrument operation.

Matrix Spike (MS) - A matrix spike provides information about the effect of the sample matrix on the digestion and measurement process. The spike is added prior to digestion. Matrix spikes are analyzed for each matrix for each analytical batch of 20 samples. Analyte spiking levels are specified in GEL's digestion SOPs. If the matrix spike recovery falls outside the specified control limits, the data associated with that spiked sample must be flagged.

Sample Duplicate (SD) and Matrix Spike Duplicate (MSD) - Sample duplicates and matrix spike duplicates are analyzed for each matrix within an analytical batch of samples. The results of the duplicate analyses serve as an indicator of the precision of the method and the sample results.

# C. Quality Control for Analysis of General Inorganic Parameters

# Method Performance OC for Analysis of General Inorganic Parameters

Method Blank (MB) - The analysis of a method blank provides the laboratory and the regulatory agencies a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. At least one method blank of similar matrix as the samples must be prepared and analyzed with each analytical batch of samples. If the concentration of the method blank is above the reporting limit, the analysis is terminated, the problem identified and corrected, and the samples reanalyzed.

Sample Duplicate (SD) and Matrix Spike Duplicate (MSD) - Every batch of 10 samples for each general inorganic parameter must include one sample prepared and analyzed in duplicate. If the results of the two samples do not fall within the specified control limits, the batch is considered suspect and if an exact problem cannot be determined, the entire batch is reanalyzed.

Matrix Spike (MS) - Every batch of 10 samples for each general inorganic parameter must include one sample spiked with a known concentration of the analyte that is being analyzed. If the results of the spike does not fall within the specified control limits, the samples are either flagged for matrix interference or the entire batch is reanalyzed.

Initial Calibration Verification - Initial calibration verification (ICV) is performed immediately following calibration by analyzing an independent solution to verify and document the accuracy of the initial calibration for each analyte. When ICV results exceed the specified control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) - An initial calibration blank (ICB) is analyzed each time the instrument is calibrated. A continuing calibration blank (CCB) is analyzed at the end of the run and at a frequency of 10% during the run. The ICB and CCB serve as a monitor of instrument drift and possible

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contamination. If the ICB or CCB result is greater than the reporting limit, the analysis is terminated, the problem identified and corrected, and the instrument recalibrated.

Continuing Calibration Verification (CCV) - To assure calibration accuracy and monitor instrument performance during each analysis, a continuing calibration verification (CCV) standard is analyzed after every 10 samples and at the end of the analysis. The analyte concentration must be at or near the mid-range level of the calibration. When the CCV results exceed the specified control limits, the analysis is terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

# D. Quality Control for Analysis of Semi-Volatile Organics

# Instrument Performance OC for Analysis of Semi-Volatile Organic Parameters

Tune - Prior to analysis of any standards, blanks, or samples by GC/MS, the ion criteria for decafluorotriphenylphosphine (DFTPP), established in the EPA Method 625 or SW-846 Method 8270, must be met. The DFTPP tune must be successfully demonstrated every 12 hours of analysis time.

<u>Initial Calibration</u> - Five-point calibrations at designated concentrations are required prior to any analysis of blanks or samples to define the dynamic range of each analytical instrument.

System Performance Check Compounds (SPCC) - This standard is analyzed after initial calibration of the GC/MS and must meet specific requirements in the analytical methods.

<u>Calibration Check Compounds (CCC)</u> - This standard is analyzed after initial calibration of the GC/MS and must meet specific requirements in the analytical methods.

Continuing Calibration Verification (CCV) - Every 12 hours of analysis, a continuing calibration verification (CCV) (at a designated concentration according to EPA Method 625 or SW-846 Method 8270) must be analyzed following a successful DFTPP tune.

## Sample OC for Analysis of Semi-Volatile Organic Parameters

Method Blank (MB) - Method blanks are analyzed for every 20 samples or where applicable, in smaller batches of samples. If phthalates are being analyzed, they must not appear in the method blank at greater than five times the required detection limit. All other semi-volatile target compounds must not be present in the blank at greater than the reporting limit.

<u>Surrogate Spike</u> - Surrogate spike compounds are added to each sample prior to sample preparation at a designated concentration to determine if there is a problem caused by sample preparation, analysis, or matrix.

Matrix Spike (MS)/Matrix Spike Duplicate (MSD) - Matrix spike compounds are added to a given sample for each matrix. The recommended QC limits for relative percent

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difference (RPD) and percent recoveries of the matrix spike compounds are detailed in the specific analytical SOP.

Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) -The laboratory control sample and duplicate (LCS/LCSD) are designed to serve as a monitor of the efficiency of the analytical process. The LCS/LCSD must be analyzed on an extraction batch basis. Results of the LCS analyses must fall within established control limits. Results which fall outside the specified control limits are indicative of an analytical problem related to the extraction/sample preparation procedures and/or instrument operation.

#### Quality Control for Analysis of Volatile Organics E.

## Instrument Performance OC for Analysis of Volatile Organic Parameters

Tune - Prior to analysis of any standards, blanks, or samples by GC/MS, the ion criteria for 4-bromofluorobenzene (BFB), established in the EPA Method 624 or SW-846 Method 8240, must be met. The BFB tune must be successfully demonstrated every 12 hours of analysis time.

<u>Initial Calibration</u> - The objective in establishing compliance requirements for satisfactory instrument calibration is to ensure that the instrument is capable of producing acceptable data. The number of calibration standards is specified for each analytical method.

System Performance Check Compounds (SPCC) - This standard is analyzed after initial calibration of the GC/MS and must meet specific requirements in the analytical methods.

Calibration Check Compounds (CCC) - This standard is analyzed after initial calibration of the GC/MS and must meet specific requirements in the analytical methods.

Continuing Calibration Verification (CCV) - For GC analyses, a continuing calibration verification standard is analyzed every 20 samples and at the end of the analysis. Specific requirements for the compounds must be achieved according to the analytical method used.

## Sample OC for Analysis of Volatile Organic Parameters

Method Blank (MB) - Method blanks are analyzed for every batch. Volatile organic target compounds must not be present in the blank at greater than the reporting limit.

Surrogate Spike - Surrogate spike compounds are added to each sample prior to analysis at designated concentrations to determine if there is a problem caused by sample preparation, analysis, or matrix. If surrogate recoveries are not within the specified control limits, samples are reanalyzed to demonstrate matrix interference or achieve acceptable results.

Matrix Spike (MS) - Matrix spikes are added to a given sample for each matrix in a batch of samples. There are method specific QC limits for the matrix spike compounds. If a large deviation from these limits occurs, the method may be unsuitable for the given

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matrix. For 8000 series analyses, a matrix spike duplicate (MSD) is also prepared and analyzed.

Laboratory Control Sample (LCS) - If the MS/MSD (for 8000 series) or the MS (for 600 series) fails, a deionized water spike is analyzed.

# F. Quality Control for Analysis of GC/ECD

## Instrument Performance OC for Analysis of GC/ECD

Initial Calibration - A five point calibration is analyzed and must have a correlation coefficient of 0.995 or greater. The calibration must be verified by analyzing a standard solution from a second source. The compounds in the verification standard must be within 15% of the calibration standard.

Evaluation Mix/Degradation Mix - A standard solution containing Aldrin, Endrin, and 4,4'-DDT is analyzed every 24 hours of analysis time. The degradation of Endrin to Endrin Ketone and Endrin Aldehyde and of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE must not exceed 20%.

Continuing Calibration Verification - A continuing calibration verification standard is analyzed every 10 samples and must be within 15% of the initial calibration values.

Retention Time Windows - Retention time windows are updated every 24 hours of analysis time or after a significant retention time shift, whichever occurs first.

## Sample OC for Analysis of Pesticides

Method Blank (MB) - A method blank is analyzed for every 20 samples extracted by each of the extraction procedures. An acceptable method blank has no analyte present at a level greater than the reporting limit. If an unacceptable method blank is analyzed, all samples extracted with that method blank are re-extracted and reanalyzed.

Matrix Spike (MS) - A matrix spike (MS) analysis is analyzed for every 20 samples prepared by each extraction procedure. The MS is a replicated sample prepared in the laboratory by adding the matrix spike standard to an aliquot of a sample prior to extraction. The MS is extracted and analyzed according to the analytical SOP.

Surrogate Spike - The performance of the extraction, clean-up (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix is monitored by spiking each sample, standard, and method blank with surrogate compounds. If the surrogate recovery does not fall within the specified control limits, corrective action as described in the appropriate analytical SOP is taken.

Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD) - A laboratory control sample and laboratory control sample duplicate (LCS/LCSD) is prepared and analyzed every 20 samples. The LCS consists of either laboratory pure water for a batch of water sample or sand for a batch of solid samples. The LCS is spiked with an SOP-defined spiking solution. If the LCS recovery does not fall within the specified control limits, corrective action as described in the appropriate analytical SOP is taken.

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#### G. Quality Control for Radiochemistry Analysis

## Instrument Performance OC for Analysis of Radionuclides

Daily Instrument Performance Check - Each day, prior to analyzing any radiochemistry samples, the analyst runs a daily instrument performance check (IPC). This IPC verifies the instrument is maintaining its calibration by falling within an acceptable tolerance. The IPC is a known source of radiation, above the instrument detection limit.

Instrument Blank - To verify the instrument is performing satisfactorily at the detection limit, an instrument blank is analyzed. The instrument blank is an empty carrier or empty chamber that is run for a set time.

## Method Performance OC for Analysis of Radionuclides

<u>Preparation Blanks</u> - The analysis of preparation blanks provides the laboratory and the regulatory agencies with a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. At least one preparation blank, consisting of deionized water must be prepared and analyzed with each batch of samples or for each 20 samples received. If the concentration of the blank is above the required detected limit, then any samples with less than 10 times the concentration level identified in the blank must be reanalyzed.

Spiked Sample Analysis - Spiked sample analysis is designed to provide information about the effect of the sample matrix on the measurement methodology. Spiked sample analyses are performed for each batch of 20 samples. A variety of factors can impact the outcome of the spike sample results; these include matrix suppression or enhancement effects, duplicate precision, contamination, and the relative levels of analyte in the sample and the spike. If the spike recovery falls outside the specified control limits, the data associated with that spiked sample must be flagged.

<u>Duplicate Sample Analysis</u> - Duplicate sample analysis is performed for each matrix and concentration level classification within a batch of samples. The results of the duplicate analyses serve as an indicator of the precision of the method and the sample results. As it is difficult to determine whether poor precision is a result of sample non-homogeneity, method inadequacies or laboratory technique, the control limits given in the EPA Protocols are to be used for flagging data and/or taking corrective action.

<u>Laboratory Control Sample Analysis</u> - Laboratory control sample (LCS) analysis is designed to provide information on the accuracy of the methodology. A LCS is a known reference material spiked into a matrix that is free from interferences (i.e.: DI water, silica sand, etc.). LCS are used for taking corrective action.

#### Additional Quality Control H.

Intralaboratory Split Samples - On occasion, it is necessary to split a single sample and identify this sample with two different identifications and submit the two samples to the laboratory as individual samples. This "blind" method of submitting the samples to the laboratory eliminates the bias that might occur if the laboratory realized that these two

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samples were the same. This type of sampling is used as a quality control check for the sample preparation, the analysis, and the quality control procedures.

<u>Statistical Process Control (SPC)</u> - Control charts are plotted for precision and accuracy for organics, metals, radiochemistry, and general inorganic parameters for:

- 1. Matrix Spikes
- 2. Blank Spikes
- 3. Duplicates
- 4. Laboratory Control Samples

These control charts are set up with both warning limits and control (action) limits. The warning limits are determined at two times the standard deviation of the recovery (accuracy) or true value (precision) for a minimum of 20 points.

For organics, metals, and general inorganic parameters, the control limits are determined at three times the standard deviation of the recovery and are updated when 20 more points are accumulated or at the beginning of each quarter, whichever happens first.

For radiochemistry, the control limits are determined at three times the standard deviation of the relative percent difference (RPD) for duplicates and are updated when 20 more points are accumulated or at the beginning of each quarter, whichever happens first. The control limits for matrix spikes and LCS's are ±25%.

Internal Standards - Internal standards are used in specific analyses according to the requirements of the analytical method. Methods where the use of internal standards for quantitation is mandatory include the analysis of base/neutral and acid extractables by GC/MS, the analysis of volatile organics by GC/MS, the analysis of some compounds by GC, and occasionally in the analysis of metals by ICP, AA, or Furnace.

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#### X. PERFORMANCE AND SYSTEMS AUDITS

GEL participates in internal and external performance evaluation studies, independent quality assessments, product quality audits, and management self-assessments.

# Performance Evaluation Studies

Performance evaluation studies are used to measure the performance of the laboratory on unknown samples. Performance evaluation samples are submitted to the laboratory as blind samples from an independent outside source or as part of the quality assurance function during internal assessment. Problems identified through participation in performance evaluation studies are investigated immediately and corrected.

GEL participates in the following performance evaluation study programs:

- The United States Environmental Protection Agency Annual Intralaboratory Water Pollution Program
- The United States Environmental Protection Agency Annual Intralaboratory Water Supply Program
- U.S. Environmental Protection Agency's Nuclear Radiation Assessment Division (EMSL-LV) Intercomparison Study.
- Department of Energy's Environmental Measurements Laboratory's (EML) Quality Assessment Program.

#### Independent Quality Assessments (Audits)

An independent quality assessment is an evaluation of quality systems and process quality. Internal quality assessments are conducted by GEL's Quality Assessor. Opportunities for improvement are identified and assistance is provided by the Audit Team to process owners for recommendations and problem resolution. Assessments or quality audits are conducted by personnel outside of the line of direct responsibility for the activity being assessed. Schedules of audit areas are prepared, annually or more frequently, based upon the significance or importance of the activity under consideration. Audits are conducted to checklists and/or written procedures. Each audit, results in an audit report which is reviewed by GEL senior management. Areas for corrective action or improvement are identified.

#### Product Quality Audits

Systematic evaluations of product quality will be conducted by the GEL Quality Team. Data accumulated on defects will be analyzed and reported to employees and management. Quality Improvement Teams (QITs) will be requested for trends and problem identification. Data analysis will include defect rates, percent defective, and statistical charts to evaluate progress.

# Management Self-Assessments

Structured assessments of quality are conducted by Group Leaders and managers at all levels. These assessments focus upon how well the quality system is working and

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identifies problems that hinder quality, safety, and environmental objectives. Senior managers will retain responsibility for actually conducting the management self-assessments. Opportunities for improvement will cause QITs to form, reporting directly back to senior management.

## Laboratory Certification

GEL participates in the following accreditation programs:

- South Carolina Department of Health and Environmental Control Certification for Drinking Water
- South Carolina Department of Health and Environmental Control Certification for Wastewater
- South Carolina Department of Health and Environmental Control Certification for Radiological Analyses
- State of California for Radiochemical Analyses
- State of Florida Certification for the Analysis of Drinking Water
- State of Florida Certification for the Analysis of Environmental Samples
- State of North Carolina Laboratory Certification Program for Environmental Samples
- State of Utah for Radiochemical Analyses (EPI -Rad only)
- State of Virginia Laboratory Certification for Inorganics and Organics
- State of Wisconsin Certification for Inorganics and Organics
- US Army Corps of Engineers (USACE) Missouri River Division (MRD)
   Validation for chemical analysis in support of the USACE Hazardous, Toxic and Radioactive Waste Program

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## XL PREVENTIVE MAINTENANCE

The preventive maintenance procedure for each instrument is described in the appropriate analytical SOP and is the responsibility of each analyst.

Each instrument has a maintenance logbook that includes the following information:

- Instrument model and serial numbers
- Instrument manufacturer
- GEL identification number
- Service person and contact information
- Instrument maintenance
- Service call documentation

The logbook also provides a baseline for what is expected of the instrument on a daily basis. If the instrument is noted during the calibration or tuning to be malfunctioning or failing, this is immediately reported to the appropriate Group Leader or Section Head. A plan for corrective action is then immediately implemented and documented. Corrective action for an instrument problem may require troubleshooting by the Group Leader, Section Head or expert for that particular instrument. Instrument problems requiring further action are reported to the GEL Instrument Service Engineer.

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# XIL STATISTICAL TECHNIQUES FOR THE ASSESSMENT OF DATA

Statistical control of analytical methods are used to determine process capability, determine out-of-control situations, and improve product and service quality. Product characteristics and product quality indicators are monitored and improved through statistical analysis. Procedures to assess precision, accuracy, and other statistical methods are described in GEL's Standard Operating Procedures and Quality Assurance Project Plans.

The majority of GEL's analytical work is performed under programs with predefined criteria for quality control or data quality objectives. In the event that GEL established control limits differ from project quality objectives, the more stringent criteria is used.

#### ACCURACY AND PRECISION

## A. Accuracy Assessment

Accuracy is the nearness of a result or the mean of a set of results to the true value. It refers to the conformity of its results to the true value of the quality characteristic being measured. Accuracy is usually expressed in terms of error or bias. Analytical characteristics which are assessed for accuracy include recoveries for matrix spike compounds, laboratory control samples, internal standards, and surrogates.

Upper and lower control limits (UCL and LCL) for accuracy are at +3 and -3 standard deviations from the mean, respectively. The upper and lower warning limits (UWL and LWL) are at +2 and -2 standard deviations.

If a result falls outside the control limits, the process is said to be "out of control". Immediate action is then taken to determine the cause of the nonconforming result. Items found to be nonconforming will be corrected prior to continuation of the process. In certain cases, as deemed necessary by the Process Control Engineer, corrective action will be taken on the nonconformances.

## B. Precision Assessment

Precision is the agreement or reproducibility of a set of replicate results among themselves without assumption of any prior information as to the true result. Precision is expressed in terms of the deviation, relative percent difference, or range. Analytical characteristics which are assessed for precision include the relative percent difference between sample duplicates, matrix spike duplicates, and laboratory control sample duplicates.

Limits for precision are set up using a series of analyses. The series consists of a set number of quality indicators analyzed in duplicate. Two duplicate determinations, A and B, are generated and the Industrial Statistic I is used. I is the absolute value of the difference for two duplicate determinations divided by their sum or:

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## I = A-BV(A+B)

The calculated I value for precision is plotted on a real-time basis. Upper warning and control limits are based on +2 and +3 times the standard deviation. The lower control limit for precision for two duplicate determinations is zero.

If a result falls outside the control limits, the process is "out of control". Immediate action is taken to determine the cause of the nonconforming result. Items found to be nonconforming will be corrected prior to continuation of the process. In certain cases, as deemed necessary by the Process Control Engineer, corrective action is taken on these nonconformances.

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#### XIII. NONCONFORMANCE AND CORRECTIVE ACTION

The process of analyzing samples includes following quality guidelines that ensure the accuracy and precision of final data. Documentation of conditions adverse to quality and the implementation of solutions to such conditions are handled through the nonconformance and corrective action systems following GEL SOPs GL-QS-E-004 for Nonconformance Identification Control, Documentation, Reporting and Dispositioning and GL-QS-E-002 for Conducting Corrective Action. A master log is initiated for all nonconformances and correction actions reported on a daily basis. An example of the Nonconformances, Corrective Action and Variances Master Log is provided in Appendix 10.

#### A. Nonconformance

Nonconformances are routinely identified and dispositioned through the Company-Wide Nonconformance Report form when established quality criteria have not been met. An example of this form is provided in Appendix 11. The processing of a nonconformance means that acceptable solutions to the immediate problem have been implemented, but that the underlying root causes of the problem have not been identified and rectified. When a problem or situation becomes repetitive or costly, corrective action is warranted.

Nonconformances may be issued from the laboratory concerning quality control data that yield results outside of established acceptance limits. Instrument processes such as failure of initial and continuing calibrations, background responses interfering with analyte readings, and instrument malfunction or failure are also considered nonconformances. Improperly preserved samples, old or contaminated calibration standards, and faulty laboratory materials are identified and dispositioned as nonconformances.

#### B. Corrective Action

When repetitive and/or costly nonconformances, conditions severely adverse to the quality of final data, or potentially hazardous situations are identified, corrective action is initiated by completing a Company-Wide Corrective Action Request and Report form. An example of this form is provided in Appendix 12. The corrective action is assigned a code and its progress tracked by the Quality Department. An example of the Tracking Status for Correction Action form is provided in Appendix 13. A Corrective Action Team is formed to problem solve and is empowered to implement corrective action.

After the Corrective Action Team has subjected the problem to root cause analysis, verified that the solutions will permanently solve the problem, implemented the corrective action, and outlined a plan to prevent reoccurrence of the problem, the solutions are reviewed and approved by the Quality Assurance Manager.

The Quality Department provides Senior Management with monthly nonconformance summaries and quarterly corrective action summaries. The monthly nonconformance summary illustrates the total number of nonconformances, the departments from which the nonconformances originated, and the frequency of the

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nonconformance reoccurrence. These reports are then distributed to Section Heads and Group Leaders to be used in continuous process improvement efforts.

Corrective action summaries are provided quarterly to Senior Management. These summaries keep Senior Management informed of the status of corrective actions. Senior Management in turn assures adherence to the corrective action system.

# C. Corrective Action Resulting from On-Site Audits

The nature of the on-site audit for the certification programs is to detect deficiencies in the overall operation of the laboratory, including methodologies, instrumentation, personnel, and data handling/storage. Findings identified through an on-site audit are corrected to meet the full requirements of the auditing agency.

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# XIV. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Monthly quality reports are presented to Senior Management to keep them informed of the quality of the data generated by the laboratory. The reports are presented by the Quality Assurance Manager and discussed in meetings with the senior managers of GEL. Items which are discussed include:

- Quality assurance problems requiring corrective action and recommended solutions.
- · Results of performance evaluation samples analyzed by the laboratory.
- Results of systems audits conducted by clients and/or regulatory agencies.
- Results of data validation audits conducted by clients and/or regulatory agencies.
- Results of internal systems audits conducted by GEL Quality Department.
- Results of data validation audits conducted by GEL Quality Department.
- Monthly updates and summaries of nonconformance and corrective action reports.
- Updates, summaries, and corrective actions from inter-departmental Corrective Action Teams.

If an analytical or engineering project or contract requirement specifies a Quality Assurance/Quality Control Report. GEL submits the data and report to the client or the regulatory agency in the required format.

#### REFERENCES

DOE Order 5700.6C, Quality Assurance, U.S. Department of Energy, August 21, 1991.

Regulatory Guide 4.15, Revision 1, U.S. Nuclear Regulatory Commission, February 1979.

10 CFR 50, Appendix B, U.S. Code of Federal Regulation.

NOA-1 Quality Assurance Requirements for Nuclear Facilities, American National Standards Institute, NOA-1, 1989.

Handbook of Quality Control for the Analytical Laboratory, James P. Dux, Ph.D., Van Nostrand Reinhold Company, New York, New York, 1986.

State of Florida Department of Environmental Regulation Guidelines for Preparing Quality Assurance Plans, DER-QA-001/85.

Handbook for Analytical Quality Control in Water and Wastewater, EPA-600/4-79-019.

Procedures Manual for Groundwater Monitoring at Solid Waste Disposal Facilities, EPA-530/SW-611, Revision 1, January 1986.

Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, U.S. Environmental Protection Agency, 3rd Edition, SW-846, 1992.

Maximizing The Statistical Performance of Groundwater Monitoring Systems, U.S. Environmental Protection Agency, Underground Resource Management, Inc. Ross and Elton, 1981, Austin, Texas.

National Handbook of Recommended Methods for Water Data Acquisition, Office of Water Data Coordination, Geological Survey, U.S. Department of Interior, 1977.

Microbiological Methods for Monitoring the Environment, Water and Wastes, EPA 600/8-78-017, December, 1978.

Groundwater Sampling Methods, Groundwater Protection Division, Bureau of Water Supply and Special Programs, SC Department of Health and Environmental Control, October, 1981.

Sampling of Water and Wastewater, U.S. Department of Commerce National Technical Information Service, PB-272664, August, 1977.

Preparation of Soil Sampling Protocol: Techniques and Strategies, U.S. Department of Commerce, National Technical Information Service, PB-206979, May 1983.

Pesticide Residue in Water, EPA-430/1-74-012, September, 1974.

Methods for Chemical Analyses of Water and Wastes, EPA-600/4-79-020, March, 1979.

Standard Methods for the Analysis of Water and Wastewater, 17th Edition.

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40 Code of Federal Register (CFR), Part 136, October 1984, Part VIII, EPA 600 Series Methodologies for the Analysis of Organic Contaminants.

Quality Control in Remedial Site Investigations, ASTM/STP 925, Fifth Volume, May 1986.

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## XV. SKILLS AND TRAINING

Employee training is conducted following GEL SOP's GL-HR-E-001 for Assessment of Employee Training Needs and GL-HR-E-002 for Employee Training. The SOPs describe needs assessment, qualification (following training), and training documentation. Training may involve in-house training; university/college courses; professional and trade association conferences, seminars, and courses; and/or one-on-one training. After receiving training, personnel are qualified by a method appropriate to the type of training including, but not limited to, attendance records, observation of proficiency, audits or tests. Following training for all laboratory analyses, analysts/technicians must demonstrate an observable level of proficiency and successful analysis of samples of known concentrations before being qualified to perform the analysis.

Following are resumes of key personnel at GEL and EPI.

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## JAMES T. CHAMBERS

Title

Laboratory Manager

Education

BS Business Administration, Troy State University, 1977 United States Air Force Electronic Training, 1973-1974 Electronic and Hardware Theory Courses for Quadrupole Mass

Spectrometers, Finnigan Corporation

System Operation and Theory of FTIR Instrumentation, Nicolet

Corporation

Zeeger Miller Frontline Leadership, 1989

Executive Management Training, Harvard Business School

Leading for Total Quality Performance

Experience and Qualifications

General Engineering Laboratories: 1990 - Present

Responsible for overall technical management of environmental laboratory, including direct supervision of over 60 chemists, degreed analysts, technicians, and laboratory support personnel. Responsible for cost control, budgeting, scheduling, and responding to clients' specific analytical needs. Analytical responsibilities include supervision of sample preparation procedures, wet chemical analyses, and analysis of organic compounds using GC and GC/MS instrumentation and approved Environmental Protection Agency reference methodologies.

CompuChem Laboratories: 1984 - 1990

Director of Technical Services (1990) - Responsible for preparation of laboratory for changes in the EPA-90 Statement of Work. This task involved major changes in the extraction procedures of liquid samples, the choice and set-up of a liquid-liquid system to meet production needs, and selection of new mass spectrometers and their implementation into normal production mode. Served as Project Manager for automation and technology project to improve data processing of the environmental product.

ChemWest, CompuChem Laboratories: 1989 - 1990

Operation Manager - Responsible for daily operations of laboratory, with particular emphasis on process backlog, quality of product to customer, and on-time percentage rate.

CompuChem Laboratories: 1988 - 1989

Manager of Organic Laboratory - Responsible for overseeing Extraction, GC, and GC/MS Departments and supervision of a staff of three managers, 30 chemists, and 28 technicians. Implemented a Management Training Program for section leaders. Maintained production goals and increased on-time rate to customer through implementation of a more visible tracking program.

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JAMES T. CHAMBERS

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CompuChem Laboratories: 1984 - 1987
Manager of Laboratory Instrumentation - Responsible for organization and training of a technical work force to provide preventative and unscheduled maintenance for \$5,000,000 of analytical instrumentation.

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#### HEYWARD H. COLEMAN

Title

President, Environmental Physics, Inc.

Education

BA Physics and Math, University of the South, 1966

MA Nuclear Physics, Duke University, 1968

MBA, Harvard Business School, 1975

Experience and Qualifications

General Engineering Laboratories and Environmental Physics

1991 - Present

Established long range planning at GEL. Organized Environmental Physics, Inc. Currently concentrating efforts on expanding capabilities of Environmental Physics to provide a full range of radiological analyses for its customers.

Maritrans GP Inc. 1981 - 1990

Executive Vice President, Director, and part owner of a large master limited partnership in the oil transportation industry. Led project that enabled Maritrans to go public through a New York Stock Exchange offering. Extensive experience in long range planning, finance, administration, and marketing.

Southern Natural Gas Co. 1977 - 1980

Executive Assistant to President of the principal interstate natural gas transmission company in the Southeast. Coordinated complex engineering projects. Established and ran a long range planning department.

Sonat Inc. 1975 - 1980

Corporate Planner in a \$3 billion energy holding company serving the oil and gas industry. Responsible for preparing long-range plans and for finding and evaluating acquisition candidates and new business opportunities.

United States Navy 1968 - 1973

Graduated from Nuclear Power School and Nuclear Prototype Training. Engineering division officer aboard a polaris missile submarine.

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GEORGE C. GREENE, P.E., Ph.D.

Title

Chief Executive Officer

Education

BS Chemical Engineering, University of Florida, 1967 MS Chemical Engineering, Columbia University, 1969 Ph.D. Chemical Engineering, Tulane University, 1973

Specialized Training

Various Environmental Protection Agency and South Carolina Department of Health and Environmental Control Environmental Seminars (1981 - Present)

Registrations/ Certifications Licensed Professional Engineer

South Carolina Registration No. 9103 North Carolina Registration No. 17511 Georgia Registration No. 19583

Professional Qualifications

General Engineering Laboratories: 1981 - Present

Dr. Greene is responsible for the development of and technical administration of analytical and environmental consulting services at General Engineering Laboratories. He has extensive experience in a wide range of projects involving investigation, design and implementation of corrective action at soil and groundwater contamination sites. He has prepared RCRA Part A and Part B permit applications and has designed and supervised closures for RCRA and CERCLA sites. Dr. Greene is also experienced in the formulation and implementation of site health and safety plans.

With the President of the organization, Dr. Greene has headed up a team which has brought professional management training into the organization to create a quality workplace environment which will enable the company to attract and retain highly qualified professionals. He leads a senior management team in implementing total quality management procedures aimed at ensuring that the quality of the company's analytical and consulting services meet stringent criteria of excellence.

- Analytical responsibilities include supervision of and providing advice to professional staff with responsibilities for sample management and analysis, methods development, quality control, and instrument maintenance.
- Technical responsibilities include review and interpretation of analytical results, instrument procurement, client support and follow-up.
- Engineering responsibilities include supervision and advice to engineering and hydrogeology staff with responsibilities for RCRA and NPDES permitting, monitoring, and corrective action, development of groundwater and surface water monitoring programs, execution of groundwater studies, development of hazardous waste cleanup and disposal criteria,

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## GEORGE C. GREENE, P.E., Ph.D.

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preparation and implementation of remedial action programs at RCRA and CERCLA sites, preparation and implementation of comprehensive environmental audits, industrial wastewater evaluation, waste treatment process development, modification, and design.

#### Presentations

"Past, Present, and Future Environmental Regulation in the United States," Southern Rubber, 1988.

"Hazardous Waste: What It Is and How It's Handled," Southern Rubber Group, Inc., Best Paper of the Year, 1989

"Managing and Disposing of Waste in Today's Regulatory Environment," HazMat South, 1991

"A Guide to Selecting Laboratory Services: The Search for Quality," Southern Rubber, 1992

Various Presentations on Environmental Issues to Industrial and Municipal Clients and Regulatory Agencies, 1981 - Present

Various Environmental Consulting and Analytical Training Programs and Special Courses (1981 - Present)

#### Publications

1969. Oxygenation of Sodium Sulfite and Blood. MS Thesis, Columbia University, 68p.

1973. Control and Simulation of a Distributed Parameter System: The Tubular Chemical Reactor, Ph.D. Dissertation, 359p.

1973 - 1977. Various Technical Reports on Salt Water Desalination, Coal Gasification, and Coke Oven Desulfurization.

1977 - 1981. Various Technical Reports on Design and Optimization of Residium Conversion Processes.

1984. General Engineering Laboratories Analytical Procedures Manual, 140p.

1985. General Engineering Laboratories Ouality Control in Environmental Testing and Hazardous Waste Characterization Manual, 29p.

1985 - 1986. Updates to General Engineering Laboratories Analytical Procedures Manual, 140p.

1990. General Engineering Laboratories Technical Writing Manual, 37p.

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GEORGE C. GREENE, P.E., Ph.D.

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1991. Managing and Disposing of Waste in Today's Regulatory Environment," HazMat South '91 Proc., 185-190.

1991. TCLP: Where Are We and What Lies Ahead? Am. Environ. Lab. December, 1991, 9-14.

1991. General Engineering Laboratories Project Management Handbook, 11p.

Professional Affiliations American Institute of Chemical Engineers
American Society for Testing and Materials
Water Pollution Control Association of South Carolina
American Chemical Society
South Carolina Laboratory Management Society
American Council of Independent Laboratories
Who's Who Registry - Platinum Edition

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## MOLLY F. GREENE

Title

President

Education

BA Liberal Arts, Mississippi University for Women, 1969 EPA Sponsored Seminar on Solid Waste Testing and Quality

Assurance, 1985, 1986 and 1989

EPA sponsored seminar on Groundwater Monitoring DHEC sponsored seminar on Underground Storage Tank

Regulations

Perkin Elmer - LIMS Management Course, Norwalk, Connecticut,

1985

Leading for Total Quality Performance

Licenses

Organizations and Water Pollution Control Association of South Carolina (WPCA),

Chairperson, Laboratory Affairs Committee, 1990

South Carolina State Chamber of Commerce American Council of Independent Laboratories

South Carolina Laboratory Management Society (SCLMS)

Business Education Partnership Program

Rotary Club, Board of Directors United Way, Board of Directors

Who's Who Worldwide

Experience and Oualifications

General Engineering Laboratories: 1981 - Present

Responsible for business administration of environmental consulting and analytical services, including Marketing and Sales, Human Resources, and GEL's Total Quality Program. Ms. Greene has extensive experience in both short term and long range planning for personnel and equipment in a full service environmental consulting firm. She is responsible for preparation of cost proposals and fee structures, contract negotiations, contract cost control, expendables inventory control.

Ms. Greene serves as a liaison between the company and client for administrative matters. Ms. Greene is responsible for personnel management, including manpower planning, forecasting, and scheduling as well as assigning adequate personnel with proper training to each project to ensure its completion in a timely and professional manner. She has a working knowledge of NPDES, SDWA, TSCA, RCRA, SDWA, Clean Air Act, UST, CERCLA, and SARA regulations.

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## GEORGE R. MCABEE, JR.

Title

Section Head - Organics

Education

BS Biology, Presbyterian College, 1975

C.N. Syd, Satisfactory completion of 4 yr. Naval Electronics

School, 1977 - 1981

Completed U.S.N.'s Main Frame Computer Maintenance

School, 1982

Attended several Naval Electronics System's Schools, 1984

Laboratory Inventory Seminar, Atlanta, Ga., 1989

Perkin Elmer Laboratory Information Management System

School, Norwalk, Cn., 1989

Tracor GC Chromatography Course, Austin, TX., 1990

Tekmar Course on VOL Purge and Trap Analysis, Cincinnati, Oh.,

1990

Geology and Hydrogeology of the USA, In-house training, 1990 Perkin Elmer/Nelson 2600 Data System, In-house training, 1990 ACS Capillary Chromatography short course, In-house training,

1990

Experience and Qualifications

General Engineering Laboratories: 1989 - present

Section Head - Organics (1993 to Present) - Responsible for the management of the Organics Section of the Laboratory. Reports directly to the Lab Manager, and facilitates communication between Client Representatives and analysts. Coordinates analytical projects, manages the training of personnel, and implements and monitors technical and quality procedures.

Group Leader - Volatile Organics (1991 - 1993) - Responsible for management of Volatile Organics Section of the Laboratory, ensuring that all analytical procedures are completed in strict accordance with approved methods. Coordinates workload and sample status. Oversees training of all new chemists and technicians. Meets daily with Laboratory Manager, Customer Service Group Leader and others to schedule incoming and existing work.

Instrumentation Technician (1989 - 1991) - Responsible for routine maintenance and repair on the following instrumentation: Hewlett Packard and Finnigan Mass Spectrometers, Tracor ECD's, Tracor PID's equipped with Hall Detectors, Perkin Elmer ICP, Zeeman 5100 Graphite Furnace and Thermal-Jarrell Ash ICP. In the wet chemistry, responsible for the maintenance of Dionex Ion Chromatograph, PE FTIR, TRAACS 800 and other various instruments. Responsible for keeping records on all major expenditures on service contracts, parts, labor, etc. Coordination of all repair activities involving outside vendors. Trains analyst on the proper maintenance of their instruments.

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GEORGE R. MCABEE, JR.

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US Naval Shipyard: 1976 - 1989

1988 - Electronics Group Quality Supervisor responsible for tracking all on-going projects within group. Provided weekly management reports on group's performance. Training of other supervisors in the quality improvement area.

1986-1988 - Electronic Systems Technician responsible for the completion, on time and within budget, of several naval electronics systems. Supervisory responsibilities included interviewing, hiring and scheduling for a staff of 25.

1985 - Electronic Systems Technician responsible for the completion, on time and within budget, of several naval electronics systems.

1983 - Senior Technician responsible for trouble shooting at least one entire naval electronic system and for the coordination of civilian personnel with naval personnel to ensure proper operation of equipment.

1978 - Electronics Technician responsible for the troubleshooting of basic naval electronic equipment.

1976 - Radiological control and radioactive material technician responsible for the proper transport of radioactive materials, as well as, the monitoring of people and equipment.

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## ROBERT L. PULLANO

Title

Quality Assurance Manager

Education

BS Marine Science/Biology, Southampton College of Long Island

University, 1984

Organizations and Licenses

American Society for Quality Control

Water Pollution Control Association of South Carolina (WPCA)

Experience and Qualifications

General Engineering Laboratories: 1988 - Present

Quality Assurance Manager (1993 - present) - Responsible for the management of the Quality Assurance Section including supervision and training of quality assurance personnel. Has responsibility for formulation of quality systems and policy and the implementation and continued improvement of the quality system.

Process Control Leader/Group Leader of Ouality Assurance Group (1991-1993) - Responsible for training and development of technical staff and coordinator of staff for analytical report generation. Established quality standards and indicators which aid in the measurement of performance and targeted processes for improvement using indicators of process quality, client satisfaction surveys, and process capability studies. Facilitated and trained process improvement teams. Coordinated Nonconformance and Corrective Action Reporting.

Ouality Control Officer/Group Leader (1989 - 1992) - Has responsibility for formulation of QA/QC policy and the implementation and continued development of total QA/QC program. Coordinate State and Federal audits of laboratory performance together with development of analytical methodology. Conduct daily laboratory data reviews, compilation and evaluation assuring Chain of Custody and Certificate of Analysis documentation. Responsible for training and development of technical staff. Implement corrective action and/or procedures when indicated to ensure that all phases of data production and reporting are performed as required thus meeting client needs as well as QA/QC guidelines.

GC Operator (1988 - 1989) - Responsible for analysis of routine and unknown organic compounds using approved EPA reference methods for GC instrumentation. These include analysis of Solvent Scans particularly F-listed wastes as well as analysis of trace level pesticides and herbicides by electron capture and analysis of volatile organics by PID/Hall.

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ROBERT L. PULLANO

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GHR Analytical: (1985 - 1988)

Senior environmental technician responsible for supervision of organic analysis. Involved overseeing staff execution of daily analysis workloads, weekly report deadlines, and QA/QC requirements. Responsible for execution and analysis of water and soil for Volatile Aromatics, Volatile Halocarbons, Trihalomethanes, PCBs, Pesticides and Herbicides by gas and liquid chromatographic techniques. Developed methodology for "in-field" analysis of volatile organic compounds at hazardous waste sites. Has a working knowledge of Superfund sample collection, handling, analysis, and quality control criteria.

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## KENNETH A. RODES

Title

Section Head - Inorganics

Education

BS Biology, Presbyterian College, 1986

Experience and Oualifications

General Engineering Laboratories: October 1991 - Present

Section Head - Inorganics (1993 to Present) - Responsible for the management of the Inorganics Section of the Laboratory. Reports directly to the Lab Manager, and facilitates communication between Client Representatives and analysts. Coordinates analytical projects, manages the training of personnel, and implements and monitors technical and quality procedures.

Group Leader, Metals (1992 - 1993) - Responsible for ensuring that all metals analyses are performed in strict accordance with approved analytical procedures and within the allowed holding times. Responsible for the first level of quality control before the analytical data is released to the Quality Control Group for further QA/QC review.

Team Task Leader. (1991 - 1992) - Responsible for performing and directing four other analysts in performing sample analyses. Performs operator maintenance on analytical instruments and preparation of standard solutions. Performs Quality Assurance reviews of all data produced and enters final data into LIMS. Trains analysts. Maintains and orders parts and supplies. Coordinates with customer service for special handling of samples.

### February 1991 - October 1991:

Responsible for accurately determining and recording elemental concentrations in environmental samples using Perkin Elmer Plasma II ICP and Thermo Jarrell Ash ICAP 61E. Prepares working standards, calibrates instrument and maintains quality control documentation in accordance with EPA methodologies. Responsible for operator level maintenance of instrument.

## <u>U.S. Army: 1989 - 1991</u>

Signal Company Operations and Training Officer responsible for the training and operations for a 130 person organization. Responsible for providing telecommunications service to a five city area.

## <u>U.S. Army: 1987 - 1989</u>

Regimental Communications Platoon Leader responsible for leading and directing the activities of 30 personnel.

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## JAMES B. WESTMORELAND

Title Laboratory Manager, Environmental Physics, Inc.

Education Bachelor of Science in Chemistry, Bob Jones University, 1982

Experience and Qualifications

Environmental Physics. Inc.: 1993 - Present
As Laboratory Manager for Environmental Physics, Inc., Mr.
Westmoreland is responsible for all laboratory and related operations. This includes the personnel management, production of chemical analyses, method development activities, nuclear instrumentation, radiation safety, radioactive waste disposal, associated quality control and support of business development.

General Engineering Laboratories: 1991 - 1993

As Radiochemistry Group Leader/Radiological Safety Officer Mr. Westmoreland was responsible for management of all personnel and radiochemistry activities in the laboratory. This included sample analysis activities as well as Radiation Safety activities such as maintaining the radioactive material inventory, performing laboratory inspections, training, updating SOP's for handling radioactive material, and ensuring laboratory compliance with state and federal regulations.

Chem-Nuclear Laboratory Services/Clemson Technical Center, Anderson, SC: 1987 - 1991

As Senior Analytical Radiochemist/Radiation Safety Officer Mr. Westmoreland was responsible for supervision of radioanalytical technicians duties as Radiation Safety Officer, maintaining radioactive material license.

Training

Alpha Spectroscopy Sample Preparation Techniques: On site training at Oak Ridge National Laboratory on various techniques available for spectroscopy mounting.

Gamma Spectroscopy Interpretation: On site training at The Nucleus Inc. on software and specifics for interpreting gamma spectroscopy data.

Radiation Safety Officer: Forty hour training on establishing and maintaining an effective radiation safety program by Radiation Safety Associates, Hebron, Connecticut.

Radioactive Waste Packaging and Disposal Workshop: Forty hour training on Department of Transportation and Nuclear Regulatory Commission regulations on shipping and disposal of radioactive material. Chem-Nuclear Systems, Inc., Columbia, South Carolina.

Nuclear Chemistry: Advanced principles of radiochemistry with emphasis on separations and nuclear instrumentation. 1992. College of Charleston.

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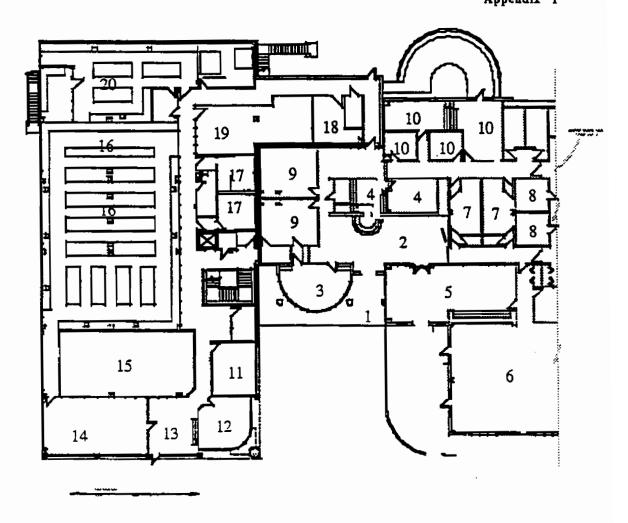
## JAMES B. WESTMORELAND

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Publications

1992. "Radium-228 in Natural Waters Via Extraction Chromatography" 38th Annual Convention on Bioassay, Analytical, Environmental, and Radiochemistry

1993. "A Simple Self-Absorption Correction for Gamma Ray Counting of Soils and Sediments" 39th Annual Conference on Bioassay, Analytical, Environmental and Radiochemistry



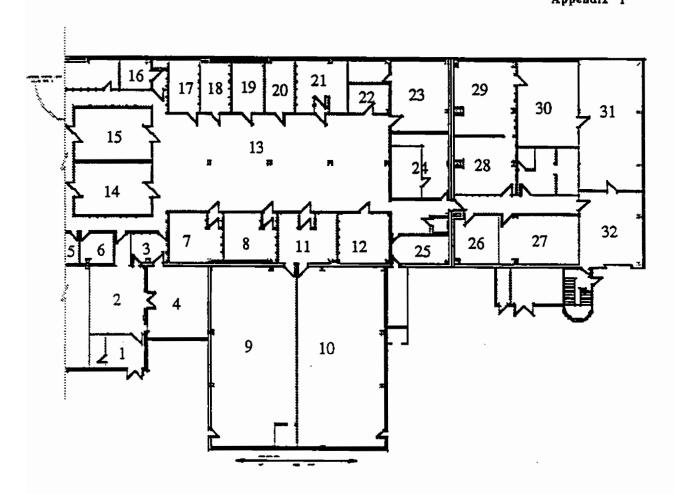
## GENERAL ENGINEERING LABORATORIES, INC., & AFFILIATES

# Facility - North Wing

- 1 Entrance
- 2 Lobby3 Conference Room
- 4 Publications
- 5 Library
- 6 Laboratory Project Mgrs
- 7 Rest Rooms
- 8 Sample Storage
- 9 Computer Services
- 10 Quality Group

- 11 18 Environmental Physics, Inc.
- 11 EPI President's Office
- 12 Conference Room
- 13 Break Room
- 14 Administration
- 15 Count Room
- 16 Radiological Prep Laboratory
- 17 Rest Rooms
- 18 Sample Storage
- 19 20 High Purity Standards, Inc.

GEL Executive Offices, Human Resources, Accounting, Marketing, and training room are located on the second floor of the North Wing.



# GENERAL ENGINEERING LABORATORIES, INC. & AFFILIATES

## Facility - South Wing

- Sample Login
- 3 Ambient Sample Storage
- Sample Storage Cold
- 5 Laboratory Manager
  6 Utility *
- Air Laboratory
- 8 Dioxin Laboratory
- 9 Extractable Organics Laboratory
- 10 Volatile Organics Laboratory
- 11 Organics Laboratory Offices
- 12 Biological Laboratory
- 13 General Chemistry
- 14 Organics Preparation Laboratory
- 15 Organics Preparation Laboratory *
- 16 Utility *
- 17 Utility *

- 18 TOC Laboratory *
- 19 Automated Chemistry Laboratory
- 20 TOX Laboratory *
- 21 Hazardous Waste
- 22 Standards Preparation Laboratory
- 23 Data Processing *
- 24 Section Heads/Group Leaders *
- 25 Utility *
- 26 Uninterruptable Power Supply (UPS)
- 27 Utility *
- 28 Inorganics Preparation Laboratory *
- 29 TCLP/Mercury Laboratory *
- 30 ICP/Graphite Furnace Laboratory *
- 31 Sample Residue Storage and Disposal
- 32 High Bay Storage

GEL Environmental Consulting Division and additional training room are located on the second floor of the South

* Tentative floor plan for completion in July 94.

General Engineering Laboratories, Inc.

Quality Assurance Plan

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Appendix 2

APPENDIX 2 Sample Storage and Preservation Requirements

Parameter	Container ¹	Preservation	Holding Time ²
Inorganics			
Acidity	P. G	4℃	14 days
Alkalinity	P, G	4°C	14 days
Biochemical Oxygen	P, G	4°C	48 hours
Demand (BOD)	-, -		75 25 22 2
Bromide `	P, G	None	28 days
Chemical Oxygen Demand (COD)	P, G	4°C, H₂SO₄ to pH<2	28 days
Chlorine by Bomb	P, G	None	None
Chloride	P, G	None	28 days
Color	P, G	4°C	48 hours
Conductivity	P, G	4°C	28 days
Corrosivity by pH	P	None	Immediate
Corrosivity to Steel	P	None	None
Cyanide amenable to	P, G	4°C, NaOH to pH>12,	14 days4
chlorination	-, -	0.6g ascorbic acid ³	1 - days
Cyanide, total	P, G	4°C, NaOH to pH>12, 0.6g ascorbic acid ³	14 days4
Dissolved Oxygen	G (bottle and top)		Immediate
Fixed and Volatile Solids	P, G	4°C	7 days
Flashpoint	P, G	None	None
Fluoride	P	None	28 days
Hardness	P, G	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months
Heating Value	P	None	None
Hydrazine	Ġ	HCl to pH<2	Immediate
Percent (%) Moisture	P	4°C	None
Ammonia Nitrogen	P, G	4°C, H ₂ SO ₄ to pH<2	28 days
Nitrate/Nitrite	P, G	4°C, H ₂ SO ₄ to pH<2	28 days
Total Kjeldahl and Organic	P, G	4°C, H ₂ SO ₄ to pH<2	28 days
Nitrogen Odor	G	19C 7am handara	T
Oil and Grease	G G	4°C, Zero headspace	Immediate
On and Grease	G	4°C, HCl or H₂ŜO₄ to pH<2	28 days
Orthophosphate	P, G	Filter immediately, 4°C	48 hours
Total Phenols	G	4°C, H₂SO₄ to pH<2	28 days
pН	P, G	None	Immediate
Total Phosphorus	P, G	$4^{\circ}$ C, $H_2$ SO ₄ to pH<2	28 days
Residual Chlorine	P, G	None	Immediate
Salinity	P	None	28 days
Specific Gravity	P	4°C	7 days
Sulfate	P, G	4°C	28 days
Sulfide	P, G	4°C, add ZnAce and NaOH to pH>9	7 days

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Parameter	Container	Preservation	Holding Time ²
Sulfite Sulfur by Bomb Surfactants	P, G G P, G	None None 4°C	Immediate None 48 hours
Settleable Solid Dissolved Solid	P, G P, G	4°C 4°C	48 hours 7 days
Total Solid Total Suspended Solid Volatile Solid	P, G P, G	4°C 4°C 4°C	7 days 7 days
Total Organic Carbon	P, G P, G	4°C, HCl or H₂SO₄ to pH<2	7 days 28 days
Total Organic Halides Total Petroleum Hydrocarbons	G G	4°C, H ₂ SO₄ to pH<2 4°C, H ₂ SO₄ to pH<2	7 days 28 days
Turbidity Metals (except chromium VI and mercury)	P, G P	4°C HNO ₃ to pH<2	48 hours 6 months
Chromium VI Mercury - Wastewater and Drinking water	P P, G	4°C HNO ₃ to pH<2	24 hours 28 days
Mercury - Others Bacteriology	G	HNO ₃ to pH<2	38 days
Coliform, fecal and total Standard Plate Count Organics	P, G P, G	4°C, 0.008% Na ₂ S ₂ O ₃ ³ 4°C, 0.008% Na ₂ S ₂ O ₃	6 hours 24 hours
Base/Neutral and Acid Extrables - Water	G, teflon- lined cap	4°C	7 days for extraction 40 days after extraction for analysis
Base/Neutral and Acid Extrables - Solid and Waste	G, teflon- lined cap	4°C	14 days for extraction 40 days after extraction for analysis
Base/Neutral and Acid Extrables - Concentrated Waste	G, teflon- lined cap	None	7 days for extraction 40 days after extraction for analysis
BTEX - Solid and Sludge	G, teflon- lined septum	4°C	14 days
BTEX - Water	G, teflon- lined septum	4°C, 0.008% Na ₂ S ₂ O ₃ , zero headspace	14 days
Chlorinated Herbicides - Water	G, teflon- lined cap	4°C	7 days for extraction 40 days after extraction for analysis

Parameter	Container ¹	Preservation	Holding Time ²
Chlorinated Herbicides - Solid and Waste	G, tefion- lined cap	4°C	14 days for extraction 40 days after extraction for analysis
Volatiles - Drinking Water	G, teflon- lined septum	4°C, 0.008% Na ₂ S ₂ O ₃ ³ , zero headspace	14 days
Volatiles (excluding 2- chloroethylvinylether) - Wastewater	G, teflon- lined septum	4°C, 0.008% Na ₂ S ₂ O ₃ ³ , zero headspace, HCl to pH 2	14 days
Volatiles - Wastewater	G, teflon- lined septum	4°C, 0.008% Na ₂ S ₂ O ₃ ³ , zero headspace	7 days
Volatiles - Solid and Sludge	G, teflon- lined septum	4°C	14 days
Volatiles - Concentrated Waste	G, teflon- lined septum	None	14 days
Industrial Solvents	G, teflon- lined septum	4℃	None
Organochlorine Pesticides and PCBs	G, teflon- lined cap	4°C	7 days for extraction 40 days after extraction for
PCBs in Oil	G, teflon- lined cap	None	analysis 7 days for extraction 40 days after extraction for analysis
Dioxin	G, teflon- lined cap	4°C	7 days for extraction 40 days after extraction for analysis
Total Petroleum Hydrocarbon Radiochemistry	G, teflon- lined septum	4°C	14 days
Carbon-14 - Water and Soil	P	4°C	6 months
Gamma Isotopes - Water Gamma Isotopes - Soil Gross Alpha and Beta -	P P P	HNO ₃ to pH-2 None HNO ₃ to pH-2	6 months 6 months 6 months
Water Gross Alpha and Beta - Soil	P	None	6 months
Iodine-129 - Water and Soil	P	None	6 months
Iodine-131 - Water	P	None	6 months
Neptunium - Water Neptunium - Soil,	P P	HNO ₃ to pH-2 None	6 months 6 months
Vegetation, and Air Filters Plutonium - Water	P	HNO₃ to pH~2	6 months

Parameter	Container ¹	Preservation	Holding Time ²
Plutonium - Soil,	P	None	6 months
Vegetation, and Air Filters	•	TOLC	O IIIOMILIS
Thorium - Water	P	HNO ₃ to pH-2	6 months
Thorium - Soil, Air Filters	P	None None	6 months
Uranium - Water	P	HNO ₃ to pH-2	6 months
Uranium - Soil, Vegetation,	P	None	6 months
and Air Filters	-	110110	o monuis
Americium - Water	P	HNO ₃ to pH-2	6 months
Americium - Soil,	P	None	6 months
Vegetation, and Air Filters	-	11020	o monais
Curium - Water	P	HNO ₃ to pH-2	6 months
Curium - Soil, Vegetation,	P	None	6 months
and Air Filters	-	1.0110	отопав
Lead-210 - Water	P	HNO ₃ to pH-2	6 months
Nickel-59 - Water and Soil	P	None	6 months
Nickel-63 - Water and Soil	P	None	6 months
Phosphorus-32 - Water	P	HNO ₃ to pH-2	6 months
Phosphorus-32 - Soil	P	None	6 months
Polonium - Water	P	HNO ₃ to pH-2	6 months
Polonium - Soil	P	None	6 months
Promethium-147 - Water	P	HNO ₃ to pH~2	6 months
Promethium-147 - Soil	P	None	6 months
Radium-223- Water	P	None	6 months
Radium-224 - Water	P	None	6 months
Radium-226 - Water	P	HNO ₃ to pH~2	6 months
Radium-228 - Water	P	HNO ₃ to pH-2	6 months
Radon-222 - Water	40 ml volatile	4°C, Zero headspace	7 days
	bottle	· -,	, <b>u</b> ays
Radon-222 - Soil	P	4°C	6 months
Strontium-89/90 - Water	P	HNO ₃ to pH-2	6 months
Strontium-89/90 - Soil	P	None	6 months
Technetium-99 - Water	P	HNO ₃ to pH~2	6 months
Technetium-99 - Soil	P	None	6 months
Total Alpha Radium - Water	P	HNO ₃ to pH~2	6 months
Total Alpha Radium - Soil	P	None	6 months
Total Uranium - Water	P	HNO ₃ to pH-2	6 months
Total Uranium - Soil	P	None	6 months
Tritium - Water, Soil,	P	4°C	6 months
Vegetation, and Air Filters			<del>-</del>
Iron 55 - Water	P	NHO ₃ to pH~2	6 months
Iron 55 - Soil	P	None	6 months

P = Polyethylene; G = Glass

Samples should be analyzed as soon as possible after collection. The holding times listed are maximum times that samples may be held before analysis and be considered valid.

³ Used only in the presence of residual chlorine.

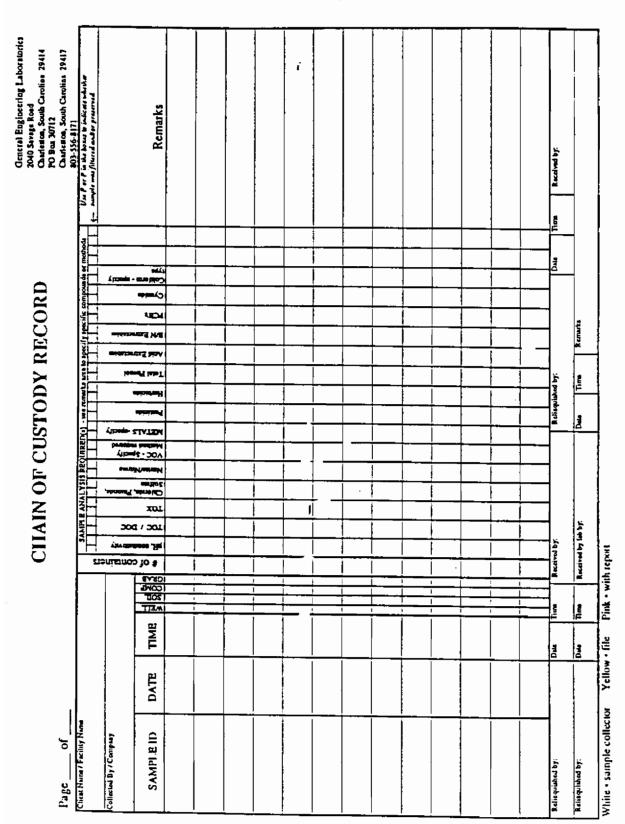
Maximum holding time is 24 hours when sulfide is present. All samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If present, remove by adding cadmium nitrate powder until a negative spot test is obtained. Filter sample and add NaOH to pH 12.

# APPENDIX 3 Field Data Information Sheet for Groundwater Sampling

Date (yr/ino/day)	1/100 ft
regradient Clear / Windy / Partly Cloudy / Wann / Cool / Hot	ration )NO
Clear / Windy / Partly Cloudy / Wann / Cool / Hol	vation )
Clear / Windy / Partly Cloudy / Warm / Cool / Hot	vation )NO
Clear / Windy / Partly Cloudy / Warm / Cool / !lot "Clear / Windy / Partly Cloudy / Warm / Cool / !lot   "C	vation )NO
Clear / Windy / Partly Cloudy / Warm / Cool / ! lot "Clar / Windy / Partly Cloudy / Warm / Cool / ! lot "C	vation )
Clear / Windy / Partly Cloudy / Warm / Cool / ! lot "Clear / Windy / Partly Cloudy / Warm / Cool / ! lot   "C	vation )
Clear / Windy / Partly Cloudy / Wann / Cool / ! lot "Clear / Windy / Partly Cloudy / Wann / Cool / ! lot "Clear / Windy / Partly Cloudy / Wann / Cool / ! lot   "Clear / Windy / Wann / Cool / ! lot   "Clear / Windy / Wann / Cool / ! lot   Windy /	vation )
Clear / Windy / Partly Cloudy / Wann / Cool / Flot 2.C	vation )
7, 00171	·
1/100/ft Locking Cep YES NO	
J 00171	
Polective Abulateut Yes	
# TWD - DGW = TIOOOTA Well Intensity Salisformary YES	•
Bal Wall Will I OW Mobels 177	
real * Standard Evacuation Volume	
Methral of Well Evucuation	
Method of Sample Collection	
Trigit Victorie of Water Removed	
ALTON A LANGE AND	
FIELD ANALISES	
VOLUMI: PURGED (galhans)	
TIMI (miliny)	
pH(S.U.)	
Sp. Cuid (paiboyem)	
Water Temp (*C)	
TURBIDITY (subjective)	
ODOR (subjective) **	
* (1) Clew (2) Slight (3) Munkrate (4) High ** (1) None (2) Faint (3) Moderate (4) Strong	
OMMIENTS/OBSERVATIONS:	ME / DATE

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APPENDIX 4
Chain of Custody Record



# APPENDIX 5

# Daily Log

Daily Log for: 22-HOR-94  Eample * Project Not Collector Description Communic CC Rubbar CL No. 70 Number Tast Machine CL No. 70 Number Machine CL No. 70 Number Tast Machine CL No. 70 Number Machine CL Number M			Daily Lo	5		
Description CCC No. PO Number Test	Daily Log	for: 22-KAR-94		Page: 1		
Description CCC No. PO Number Test						
CCC   Mambar   CCL   No.   PO   Number   Natrix   Match	•	•		Time Dace Coll P Days	Due Date	Rec'd by
### Machina   Ma		•				
9403442-01 SANTOD191 MAS SANT MARRIEDO 0715 22-MAR-94 1 14 05-AFR-94 CCB  MARRIEDO SN 16th ed. 909C TSS MARRIEDO SN 16th ed. 909C TSS MARRIEDO 0800 22-MAR-94 3 14 05-AFR-94 TAM  9403443-01 CONTOD191 PAB CONT MARRIEDO 0800 22-MAR-94 3 14 05-AFR-94 TAM  OLG MARRIEDO EPA 413.1  OLG MARRIEDO EPA 413.1  TCC MARRIEDO EPA 415.1  DOWNA-REG DELANGO EPA 413.1  10748.1  DOWNA-REG DELANGO EPA 524.2  DOWNA-REG DELANGO EPA 300.0  9403445-01 RGUY00191 AGM RGMY MARRIEDO 0845 21-MAR-94 3 14 05-AFR-94 TAM  ACID FREP MARRIEDO EPA 300.0  PAGENDO EPA 500.7  CH MARRIEDO EPA 200.7  CH MARRIEDO ASTM 2006  CU-CCP MARRIEDO EPA 200.7  CH MARRIEDO EPA 200.7  MARRIEDO ASTM 2006  CU-CCP MARRIEDO EPA 200.7			•••			
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### ### ##############################						
### ### ##############################	9403442-01	THE EAST STATE	Wasteli20	0715 22-HAR-94 3 14	05 <b>-APR-94</b>	CCB
FCMF MasteR2O SM 16th ed. 909C TSS MasteR2O SM 11.1  OLG MasteR2O SM 11.1  MasteR2O SM 11.1  MasteR2O SM 15.1  DWGA-REG DrinkR2O SM 125 21-MAR-94 3 14 05-AFR-94 TAM 94-71-1141  DWGA-REG DrinkR2O SM 100.0  9403445-01 RGWY00191 AUW RGWY MasteR2O 0845 21-MAR-94 3 14 05-AFR-94 TAM GMSP-0321  353.1  ACTO PREP MasteR2O SM 200 CD-1CF MasteR2O SM 200 CD-1CF MasteR2O SM 200.7  CH MasteR2O SM 200.7  CH MasteR2O SM 200.7  CH MasteR2O SM 200.7  M						
### PCMF						
### PASS   MasteRIO EPA 160.2  9403443-01 CONTO0191 PAB CONT						
9403443-01 COMT00191 PAB CONT						
Old Oll/Water Separator   205.1			***************************************	2.7. 2.7.2		
205.1	9403443-01	CONTOOL91 PAB CONT	WasteH20	0800 22-MAR-94 1 14	05-APR-94	TAN
OAG WASTENZO EPA 413.1 PH WASTENZO EPA 415.1  9403444-01 COMMODISI AGM COMM DENNIKZO 1325 21-HAR-94 3 14 05-AFR-94 TAM 94-71-1341  10748.1  DMVUDA-REG DFINKHZO EPA 524.2 NO3 DFINKHZO EPA 100.0  9403445-01 RGMY00191 AGM RGMY MASTENZO 0845 21-HAR-94 3 14 05-AFR-94 TAM GMSP-0321  353.1  ACID PREP WASTENZO EPA 200.7 CH MASTENZO EPA 100.7 CH MASTENZO EPA 100.7 CH MASTENZO EPA 100.7 CH MASTENZO EPA 200.7 CH MASTENZO EPA 100.7 WASTENZO EPA 100.7 WASTENZO EPA 100.7 WASTENZO EPA 200.7 WASTENZO EPA 200.0 WASTENZO EPA 2		Old Oil/Water Separator				
04G		205.1				
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### PACED PREP   WasteHZO EPA 200   PA				-		
9403444-01 CCH000191 AGM COMM DrinkH2O 1325 21-HAR-94 3 14 05-APR-94 TAM 94-71-1341  10748.1  DWUDA-REG DrinkH2O EPA 524.2 NO3 DrinkH2O EPA 300.0  9403445-01 RGWY00191 AGM RGWY MasteH2O 0845 21-HAR-94 3 14 05-APR-94 TAM GWSP-0321  353.1  ACID PREP MasteH2O EPA 200 CD-ICP MasteH2O EPA 100.7 CI MasteH2O EPA 100.7 CI MasteH2O EPA 301.1  CN-FREE MasteH2O EPA 200.7 CI MasteH2O EPA 200.7 CI MasteH2O EPA 200.7 CI MasteH2O EPA 200.7 CI-ICP MasteH2O EPA 200.7 HG-CV MasteH2O EPA 200.7 HG-CV MasteH2O EPA 200.7 HG-CV MasteH2O EPA 200.7 PR-GFAA MasteH2O EPA 150.1 PR-GFAA MasteH2O EPA 150.1 EN-ICP MasteH2O EPA 150.1 EN-ICP MasteH2O EPA 200.7  9403446-01 GOSSIO191 MAS GOSS Misc. 17-HAR-94 3 14 05-APR-94 TAM MasteH2O EPA 200.7  9403446-01 GOSSIO191 MAS GOSS Misc. 17-HAR-94 3 14 05-APR-94 TAM CRESOL-MSE Misc. EPA 8270 CDMENT VOA Misc. EPA 8270 CDMENT VOA Misc. EPA 8260 extended CRESOL-MSE Misc. EPA 3260 extended						
10748.1   10748.1   10748.1   10748.1   10748.2   EPA 524.2   100.0   10748.2   EPA 300.0   10748.2   EPA 300.7   10748.2   EPA 200.7   EPA		100	Mascanzo	25K 413.1		
DMVUDA-REG	9403444-01	COMMODISI AGN COMM	DrinkH20	1325 21-HAR-94 3 14	05-APR-94	TAH
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DMVOA-REG NO3 DrinkH2O EPA 524.2 NO3 DrinkH2O EPA 300.0  9403445-01 RGWY00191 AGW RGWY MasteH2O 0845 21-HAR-94 1 14 05-AFR-94 TAW GWSP-0321  353.1  ACID PREP MasteH2O EPA 200 CD-ICP MasteH2O EPA 135.1 CM-FREE MasteH2O EPA 135.1 CM-FREE MasteH2O EPA 200.7 CM MasteH2O EPA 150.1 CM-GREE MasteH2O EPA 200.7 HG-CV MasteH2O EPA 200.7 HG-CV MasteH2O EPA 200.7 PB-GFAA MasteH2O EPA 150.1 ZN-ICP MasteH2O EPA 150.1 ZN-ICP MasteH2O EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-HAR-94 1 14 05-AFR-94 TAW Cement Kilm Dust HCCKD21 VOA soilsGive to VOA 1st  2235.1  APP 9 ACID Misc. EPA 8270 CEMENT VOA Misc. EPA 8270 CEMENT VOA Misc. EPA 8260 extended CRESOL-HSE Misc. EPA 8260 extended		10748.1				
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		NO3	DrinkH20	EPA 300.0		
	9403445-01	economial act econ	WastaW70	0845 21-WND-94 1 14	05-199-94	#3 W
ACID PREP   WasteH2O EPA 200	1403443-01		MASCERIO	0043 44-1044-34 3 44	UJ-APK-74	IAN
ACID PREP  CD-IC?  CI						
CD-IC? WasteH2O EPA 200.7  CN WasteH2O EPA 115.1  CN-FREE WasteH2O ASTM 4282  CN-WAD WasteH2O ASTM D2036  CU-ICP WasteH2O EPA 245.1  PB-GFAA WasteH2O EPA 245.1  PB-GFAA WasteH2O EPA 245.1  PM WasteH2O EPA 150.1  ZN-ICP WasteH2O EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-MAR-94 3 14 05-APR-94 TAW  Cament Kiln Dust MCCXD23 VOA soilsGive to VOA 1st  2235.1  APP 9 ACID Misc. EPA 8270  APP 9 B/N Misc. EPA 8270  CENENT VOA Misc. EPA 8270  CENENT VOA Misc. EPA 8260 extended  CRESOL-MSE Misc. EPA 8270						
CH WasteH2O EPA 135.3  CN-FREE WasteH2O ASTM 4282  CN-WAD WasteH2O ASTM D2036  CU-ICP WasteH2O EPA 200.7  HG-CV WasteH2O EPA 245.1  PB-GFAA WasteH2O EPA 219.2  PH WasteH2O EPA 150.1  ZN-ICP WasteH2O EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-HAR-94 3 14 05-APR-94 TAW  Cement Kiln Dust HCCKD23 VOA soilsGive to VOA 1st  2235.1  APP 9 ACID Misc. EPA 8270  APP 9 8/N Misc. EPA 8270  CEMENT VOA Misc. EPA 8260 extended  CRESOL-MSE Misc. EPA 3260 extended		ACID PREP	WascaH20	EPA 200		
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CU-ICP WasteHZO EPA 200.7.  HG-CV WasteHZO EPA 245.1  PB-GFAA WasteHZO EPA 219.2  PH WasteHZO EPA 150.1  ZN-ICP WasteHZO EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-HAR-94 3 14 05-APR-94 TAW  Coment Kiln Dust HCCKDZ3 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Misc. EPA 8270  APP 9 B/N Misc. EPA 8270  CEMENT VOA Misc. EPA 8270  CEMENT VOA Misc. EPA 8260 extended  CRESOL-HSE Misc. EPA 8270						
#G-CV WasteH20 EPA 245.1  PB-GFAA WasteH20 EPA 219.2  PH WasteH20 EPA 150.1  ZN-ICP WasteH20 EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-MAR-94 3 14 05-APR-94 TAW  Coment Kiln Dust HCCKD23 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Misc. EPA 8270  APP 9 B/N Misc. EPA 8270  CEMENT VOA Misc. EPA 8260 extended  CRESOL-MSE Misc. EPA 8270						
PB-GFAA WasteH2O EPA 219.2 PH WasteH2O EPA 150.1 ZN-ICP WasteH2O EPA 200.7  9403446-01 GOSS00191 WAS GOSS Misc. 17-MAR-94 B 14 05-APR-94 TAM Cemenc Kiln Dust MCCKD23 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Misc. EPA 8270 APP 9 B/N Misc. EPA 8270 CEMENT VOA Misc. EPA 8260 extended CRESOL-MSE Misc. EPA 3260 extended						
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9403446-01 GOSS00191 WAS GOSS Misc. 17-MAR-94 3 14 05-APR-94 TAW  Cement Kiln Dust HCCKD21 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Misc. EPA 8270  APP 9 B/N Misc. EPA 8270  CEMENT VOA Misc. EPA 3260 extended  CRESOL-MSE Misc. EPA 3270		5H	WasteH20	EPA 150.1		
Cement Rilm Dust HCCRD23 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Hisc. EPA 8270 APP 9 B/N Hisc. EPA 8270 CEMENT VOA Hisc. EPA 3260 extended CRESOL-HSE Hisc. EPA 3270		ZN-ICP	WasteH20	EPA 200.7		
Cement Rilm Dust HCCRD23 VOA soilsGive to VOA 1st  3235.1  APP 9 ACID Hisc. EPA 8270 APP 9 B/N Hisc. EPA 8270 CEMENT VOA Hisc. EPA 3260 extended CRESOL-HSE Hisc. EPA 3270	0403445 5-	G0GC00101 100 0000	•••			
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APP 9 B/N Misc. EPA 8270  CEMENT VOA Misc. EPA 8260 extended  CRESOL-MSE Misc. EPA 8270		•				
CEMENT VOA Misc. EPA 8260 extended CRESOL-HSE Misc. EPA 8270		APP 9 ACID	Misc.	EPA 8270		
CRESOL-MSE Misc. EPA 8270		APP 9 8/N				
		CEMENT VOA	Misc.	EPA 8280 extended		
EGMEE-FID Misc. GC/FID						
		ECMEE-FID	Misc.	GC/FID		

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# APPENDIX 6

# Radioactive Shipment Inventory Sheet

Shipment Reference No	Date Received:			
Received By:	No. of Samples in Shipment:			
Total Sh	ipment Activity Tritium (uCi)			
	Atomic # 2-98 (uCi)			
If activity is from non to	ritium isotopes, list the isotopes below:			
Sample Information:				
Sample I.D				
Laboratory LD.				
Sample SurfaceReading (mR/hr)				
Removable alpha contamination (dpm/100cm ² )				
Removable beta contamination (dpm/100cm ² )				
Show calculations if total ships Attach applicable client informa	ment sample activity was calculated from individual samples.			
Reviewed and Approved:	Date:			

GEL QAP No.: GL-QS-B-001 - Rev. 9 Effective Date: April 8, 1994

Appendix 7

## APPENDIX 7

# Certificate of Analysis

# GENERAL ENGINEERING LABORATORIES, INC.

**Environmental Consulting and Analytical Services** 

Meeting Today's Needs With a Vision for Tomorrow

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## CERTIFICATE OF ANALYSIS

Client

Contact

œ:

Report Date: January 13, 1994

Page 1 of 1

Sample ID Lab ID

Matrix : WasteH2O
Date Collected : 01/03/94
Date Received : 01/05/94
Priority : Routine
Collector : Client

Parameter	Qualifier	Result	Units	Method	Anaiyst	Date	Time
Metals Analysis							
Mercury		0.900	ug∕l	EPA 245.1	ADF	01/10/94	1234
General Chemistry			•				
Chemical Oxygen Deman	d	164	mg/l	EPA 410.4	JBH	01/07/94	1100
Cyanide, Total		0.152	mg/l	EPA 335.3	JEN	01/10/94	1600
Chlorides		155	mg/l	EPA 300.0	BJW	01/11/94	1257
Sulfate 29 SO4		1200	mg/l	EPA 300.0	BJW	01/11/94	1310
Phenois, Total		580	ug/l	EPA 420.1	<b>JEN</b>	01/07/94	1400
Phosphorus, Total as PO4		8.00	mg/l	EPA 365_1	CEG	01/10/94	1300
The following prep procedu	res were per	formed:					
Mercury				EPA 245.1	ADF	01/06/94	1300

This data report has been prepared and reviewed in accordance with General Engineering Laboratories standard operating procedures. Please direct any questions to your Project Manager,

Analytical Report Specialist

## Explanation of Terms and Symbols

The following terms and symbols may be used in reports of analytical data:

## **Quantities**

BTU	Units of heat content expressed in British Thermal Units
°C or °F	Units of temperature expressed in degrees Celsius or degrees Eahrenheit
<	Indicates the measured value is less than the reported value
>	Indicates the measured value is greater than the reported value
NTU	Units of turbidity expressed as Nephelometric Turbidity Units

## Concentrations

ншрог/сш	Units of specific conductance expressed in micromhos per centimeter
pCi/L	Units of radioactivity expressed as picocuries per liter
mg/Kg, mg/L	Units of concentration in milligrams per kilogram for solids and milligrams per liter for liquids. Also referred to as Parts Per Million or "ppm" when the assumption can be made that the specific gravity is one (density = 1 gm/ml).
μg/Kg, μg/L	Units of concentration in micrograms per kilogram for solids and micrograms per liter for liquids. Also referred to as Parts Per Billion or "ppb" when the assumption can be made that the specific gravity is one (density = 1 gm/ml).
m\/m³	Units of concentration in milliliters per cubic meter for gases. Also referred to as Parts Per Million or "ppm."
voi%	Units of concentration expressed on a volume/volume basis (e.g., liters per 100 liters)
wt <del>%</del>	Units of concentration expressed on a weight/weight basis (e.g., grams per 100 grams)

## Bacteriological

MPN	Units of bacteriological results expressed as Most Probable Number
דאַדכ	Units of bacteriological results expressed as Too Numerous To Count

## WARRANTY AND LIMITATIONS OF LIABILITY

THE ACCURACY OF ALL ANALYTICAL RESULTS IS WARRANTED FOR THE SAMPLE AS IT IS RECEIVED BY THE LABORATORY. WARRANTY FOR THE INTEGRITY OF THE SAMPLE BEGINS AT THE TIME IT IS PLACED IN THE POSSESSION OF AUTHORIZED GENERAL ENGINEERING LABORATORIES PERSONNEL. ALL OTHER WARRANTIES, EXPRESSED OR EMPLIED, ARE DISCLAIMED. LIABILITY IS LIMITED TO THE COST OF THE ANALYSIS.

## APPENDIX 8

# Level 2 Certificate of Analysis

# GENERAL ENGINEERING LABORATORIES, INC.

Environmental Consulting and Analytical Services Laboratory Certifications E27156/27294 HC TH Meeting Today's Needs WŢ 99988779 With a Vision for Tomorrow CERTIFICATE OF ANALYSIS

Clienti

Report Date: 01/13/94

233 10120

02734 12100

Matrix: WasteW20 Description: Sample: Client ID: Sampled: 01/03/94 Received: 01/05/94 Page: 1 Project: Hanader: QL.

		**************************************											
Hereusy	Hethods	DA 245.	1		Bacchi	41415	Run	Analysts	ADT	00A:	01/10/94	TOA:	1234
•							TOP	Analyse:	ADF	90P t	01/06/94	TOP:	1300
Hecanch		0.900		0.200	uq/1								
Stological	Hethodi	ER 410.	4		Bacchi	41461	fun	Analysts	JBH	DOA	01/07/94	TOA:	1100
Chemical Oxygen Demand		164.		10.0	<b>=</b> 4/1								
Cyanide by RFA	Hethods	EPA 335.	.3		<b>L</b> atch:	41453	Run	ARALYSC:	JЕN	DOA:	01/10/94	TOA	1600
Cyanide, Total		0.152		0.0100	<b>aq/</b> 1								
Ion Chrometography	Hethod:	EPA 300.	.0		Bacch:	41488	Run	Analyst:	N.	DOA:	01/11/94	TOA:	1257
Chicrides		155.		3.75	mg/L								
Ion Chromatography	:bottoM:	EPA 300.	.0		Batchi	41446	Aug	Analyst	BJW	DOA	01/11/94	TOA	1257
Sulfate es 504		1200		75.0	<b>24/</b> 1								
RFA	Heched:	EPA 420.	.1		BACCHI	41406	Rua	Amalysc:	JD	DOA	01/07/94	TOA	1400
Phenois, Total		580.		125.	<b>uq/1</b>								
Technicon	Hethods	EPA 365.	.1		Batchs	41422	Run	Analyst	Œ	DOA	01/10/94	TOA	1300
Phosphorus, Total as P	04	E.00		1.30	<b>mg/</b> 1								

This data report has been prepared and reviewed in accordance with General Engineering Laboratories standard operating procedures. Please direct any questions to your Project Manager,

Analytical Report Specialist

## **Explanation of Terms and Symbols**

The following terms and symbols may be used in reports of analytical data:

## **Ouantities**

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*C or *F	Units of temperature expressed in degrees Celsius or degrees Eahrenheit
<	Indicates the measured value is less than the reported value
>	Indicates the measured value is greater than the reported value
NTU	Units of turbidity expressed as Nephelometric Turbidity Units

## Concentrations

mpos/cm	Units of specific conductance expressed in micromhos per centimeter
pCi/L	Units of radioactivity expressed as picocuries per liter
mg/Kg, mg/L	Units of concentration in milligrams per kilogram for solids and milligrams per liter for liquids. Also referred to as Parts Per Million or "ppm" when the assumption can be made that the specific gravity is one (density = 1 gm/ml).
μg/Kg, μg/L	Units of concentration in micrograms per kilogram for solids and micrograms per liter for liquids. Also referred to as Parts Per Billion or "ppb" when the assumption can be made that the specific gravity is one (density = 1 gm/mi).
ml/m³	Units of concentration in milliliters per cubic meter for gases. Also referred to as Parts Per Million or "ppm."
voi%	Units of concentration expressed on a volume/volume basis (e.g., liters per 100 liters)
wi%	Units of concentration expressed on a weight/weight basis (e.g., grams per 100 grams)

## **Bacteriological**

MPN	Units of bacteriological results expressed as Most Probable Number
TNTC	Units of bacteriological results expressed as Too Numerous To Count

## WARRANTY AND LIMITATIONS OF LIABILITY

THE ACCURACY OF ALL ANALYTICAL RESULTS IS WARRANTED FOR THE SAMPLE AS IT IS RECEIVED BY THE LABORATORY. WARRANTY FOR THE INTEGRITY OF THE SAMPLE BEGINS AT THE TIME IT IS PLACED IN THE POSSESSION OF AUTHORIZED GENERAL ENGINEERING LABORATORIES PERSONNEL ALL OTHER WARRANTIES. EXPRESSED OR IMPLIED, ARE DISCLAIMED. LIABILITY IS LIMITED TO THE COST OF THE ANALYSIS.

GEL QAP No.: GL-QS-B-001 - Rev. 9 Effective Date: April 8, 1994

Appendix 9

## APPENDIX 9

## Level 3 Certificate of Analysis

# GENERAL ENGINEERING LABORATORIES, INC.

Laboratory Cartifications **Environmental Consulting and Analytical Services** FL. 287156/87294 NC 233 sc 10120 TH 02934 VA. 00151 Meeting Today's Needs wz 99988779 With a Vision for Tomorrow CERTIFICATE OF ANALYSIS

Cilent:

Contact:

Report Date: 01/13/94

Sample:	Client	ĮD:			Mastell20	ð.	scription:		_		
re ject :	Manaq	mr:		Sampled:	01/03/94		Received:	01/05/94	Page:	1	
Parameter			Result	Qual	QL.	Unite	or	Surr. 1	Blank	RPD	1Rec
: Hercury		Hethod:	EPA 245.1			Batch:		-	OF DOA: 01/10		
Hercury			0.900		9,200	ue/1	Prep lil	Analyst: A	OF DOP: 01/06 -0.0920	/34 TOP: 0.4349	1300 OK
G: Bloingical		Het had s	EPA 410.4			- 4		Analyst: J	BE DOA: 61/07		
Chemical Ozygo	n Demand		164.		10.0	mq/1	1:1	, , , , ,	0.960	5.779	80.75
G: Cyanide by RF		Hethod:	EPA 335.3			Bat ch :	41453 Run	Analyst: J	DR DGA: 01/10	/94 TOA:	1600 DK
Cyanide. Total	L		0.152		0.0100	mg/1	1:1		0.00	83.19	85.5%
G: Ion Chromatog:	raphy	Hethods	EPA 300.0			Batch:	41486 Run	Analyst: \$	JW DOA: 01/11	/94 TOA	1257 OK
Calorides			155.		3.75	<b>m</b> /1	1:50		0.00	1.519	10.11
G: Ion Chromatog:	raphy	Muthodz	CPA 300.0			Batchi	41488 Run	Analyst: &	JW DOA: 01/11	/94 TOA:	1257 OK
Sulfate as 10	4		1200		75.0	mg/L	1:100		0.0200 -	4.954	55.51
GI RFA		Hethods	EPA 420.1			Betch:	11406 Run	Analyst: J	<b>EN DOA: 01/07</b>	/94 TOA	1400 OK
Phenois, Total	L		580.		125.	<b>ug/l</b>	1:25		-0.100	2.029	1204
C: Technicon		Hethodi	CPA 365.1			Batchi	41422 Run	WwTAst: C	EG OGA: 01/10	/94 TOA	: 1300 OK
thosphorus, to	otal as P	04	8.00		1.00	<b>mq/l</b>	1:10		0.0200	0.001	40.00

This data report has been prepared and reviewed in accordance with General Engineering Laboratories standard operating procedures, Places direct any questions to your Project Hanager,

Analytical Report Specialist

## **Explanation of Terms and Symbols**

. The following terms and symbols may be used in reports of analytical data:

## Quantities

BTU	Units of heat content expressed in British Thermal Units
°C or °F	Units of temperature expressed in degrees Celsius or degrees Eahrenheit
<	Indicates the measured value is less than the reported value
>	Indicates the measured value is greater than the reported value
עזע	Units of turbidity expressed as Nephelometric Turbidity Units

## Concentrations

µmhos/cm	Units of specific conductance expressed in micromhos per centimeter
pCi/L	Units of radioactivity expressed as <u>picocuries per liter</u>
mg/Kg, mg/L	Units of concentration in milligrams per kilogram for solids and milligrams per liter for liquids. Also referred to as Parts Per Million or "ppm" when the assumption can be made that the specific gravity is one (density = 1 gm/ml).
μg/Kg, μg/L	Units of concentration in micrograms per kilogram for solids and micrograms per liter for liquids. Also referred to as Parts Per Billion or "ppb" when the assumption can be made that the specific gravity is one (density = 1 gm/ml).
ml/w₁	Units of concentration in milliliters per cubic meter for gases. Also referred to as Parts Per $\underline{M}$ illion or "ppm."
vol%	Units of concentration expressed on a volume/volume basis (e.g., liters per 100 liters)
wt%	Units of concentration expressed on a weight/weight basis (e.g., grams per 100 grams)

## **Bacteriological**

MPN	Units of bacteriological results expressed as Most Probable Number
TNTC	Units of bacteriological results expressed as Too Numerous To Count

## WARRANTY AND LIMITATIONS OF LIABILITY

THE ACCURACY OF ALL ANALYTICAL RESULTS IS WARRANTED FOR THE SAMPLE AS IT IS RECEIVED BY THE LABORATORY. WARRANTY FOR THE INTEGRITY OF THE SAMPLE BEGINS AT THE TIME IT IS PLACED IN THE POSSESSION OF AUTHORIZED GENERAL ENGINEERING LABORATORIES PERSONNEL. ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, ARE DISCLAIMED. LIABILITY IS LIMITED TO THE COST OF THE ANALYSIS.

General Engineering Laboratories, Inc.

Quality Assurance Plan

GEL QAP No.: GL-QS-B-001 - Rev. 9

Effective Date: April 8, 1994

Appendix 10

# APPENDIX 10 Nonconformances, Corrective Action and Variances Master Log

Form GEL, Inc XXX	
(Rev. 6/21/93)	LOG DATE:
	MASTER LOG NO:
GENERAL ENGI NON CONFORMANCES, (	GENERAL ENGINEERING LABORATORIES, INC NON CONFORMANCES, CORRECTIVE ACTION AND VARIANCES MASTER LOG
NCR#:	CARR#:
DATE OF NCR ORIGINATION:	DATE OF CARR ORIGINATION:
NAME OF NCR ORIGINATOR:	NAME OF CARR ORIGINATOR:
SOURCE OF NONCONFORMANCE:	RESPONSIBLE PARTY OF CARR:
TYPE OF DISPOSITION*:	DUE DATE OF CARR:
DATE OF DISPOSITION:	DATE CARR CLOSED:
DATE NCR CLOSED:	
WHAT IS THE SIGNIFICANT OR REOCCURING PROBLEM?:	17:
DISCRIPTION OF ACTION TAKEN:	
*DISPOSITIONS; 1, Repair 2, Rework 3, Use As Is	2; Rework 3. Use As Is 4. Return to Vendor 5. Scrap 6. Design Change 7. Complete

## APPENDIX 11

# Company-Wide Nonconformance Report

General Engineering Laboratories		1. NCR Report No.:					
Form GEL-XXX (Rev. 11/92)	2. Page of						
(2000)		J. 347.000 110,					
COMPANY-WIDE NONCONFORMANCE REPORT COMPLETE EYERY ITEM							
		on Reverse Side)					
4. Mo. Day Yr. 5. Company Name/Department/Function 6. Material (Item) Process Product							
7. Numerical Reference Identification 8. Quality Criteria: SOP QAP or QAPJP Client Contract Purchase Document Drawing Specifications Others							
9. Supplier Name P.O. Requisition Number Subcontractor Item: Not Applicable							
10. Failed Acceptance Test Reference Number							
11. Specifications and Requirements Nonconformance Description		13. NRG Disposition					
Notice Indicate Describition							
Item No.		Item No.					
	!	i					
	]						
	-	·					
	1						
		·					
		14. NRG Printed Name & Signature Date					
		14. INCO FINICI Name & Signature Date					
ļ		List NRG Participants:					
12. Originators Printed Name & Signature I	Date	Management Review or Management Approval					
Please revie	w withi	n 24 hours of receipt.					
NCR Review & Disposition Review or Approve	al:	Corrective Action Request and Approval:					
15. Q Process Control Review	Date	18. CA Requested: Print Name and Sign Date					
16. Originator's Leader/Supervisor Name/Sig	Date	19. Corrective Action Approval Date					
17. Originator's Manager Name/Signature	Date	20. Corrective Action Number					

NCR initiator instructions: Complete items 1 through 12. Do not leave items blank. Complete or mark "N/A"

Construct an NCR number using the (Company - Department or Group - Function - Sequence number) pattern.

Companies:

General Engineering Laboratories = GEL High Purity Standards = HPS

Environmental Physics. Inc. = EPI

Departments of initiator: Analytical Services = AS Consulting Engineering = CE Quality Assurance = QA or Group Administrative Services = AD Customer Service = CS Marketing = MK None = XX

Functions of initiator:

Organic Analysis = OA Metals Analysis = MA General Chemistry = GC

Microbiological Analysis =MI Sample Preparation = SP Data Reporting = DR Library Services = LS Process Control = PC Information Systems = IS Air Quality = AQ

Project Management = PM Field Services - FS None = XX

Sequence:

-01, -02, -03 (use next available number from sequence log)

Example: NCR number = GEL-QA-DR-01

Write the correct page number and total number of pages in the NCR as you have submitted it. As the NCR passes through review and approval, it may be necessary to issue additional pages. To change, strike through, initial and date.

3. Issue the NCR as Rev Q. Later on, if it is necessary to revise the NCR, replace the NCR in its entirety and issue the correct revision number. Return "replaced" NCR to Quality Process Engineer.

Write in the date that you initiated the NCR.

Write in originator's affiliate company name, department and function in that sequence.

Example: GEL; Quality Assurance; Quality Management Systems; Example: GEL; Quality Assurance; Reporting; Example: GEL; Analytical Services; trace metals analysis.

Identify the type of nonconformance. Did it involve material, an item, a process or a finished product? Check applicable box.

- Numerical Reference Identification: This number should identify nonconforming items for disposition, such as: laboratory ID number, batch number, QC number, project number, or Purchase Order Number, solvent lot, instrument number.
- Identify the source of the quality requirements: standard operating procedure, quality assurance plan, client contract requirements, or client specific quality assurance project plan, purchase document, engineering drawing, engineering specifications, other specifications. Check as many as apply.

If the nonconformance resulted from purchased material, item, or service, write in the name of the supplier and indicate if procured from a Purchase Order, Purchase Requisition or any other procurement. Otherwise enter N/A.

- 10. If the nonconformance resulted from the failure of a quality acceptance test of a purchased product or service, assign an appropriate test reference number. This number should make the specific failure data traceable in the future. Otherwise enter N/A.
- 11. In the space provided, describe the specifications or the requirements also, the nonconformance. Example: "Solvent ordered was nanograde: received reagent grade." Example: "Ordered three cases of solvent, received only two." Because more than one nonconformance can happen with a single purchase order, itemize each nonconformance starting with the number one.

12. Initiator must print name, sign, and date NCR.

#### Nonconformance Review Group (NRG) instructions: Complete items 13 and 14.

13. In the space provided, the NRG representative describes the disposition of each nonconformance. Example: "Return to vendor"; Example: "Mark solvent to be used for glassware cleaning only;" Example: "Redraw plans to as-built condition."

14. NRG representative prints his/her name, signs and dates. List the names of other NRG participants. If dispositioning the nonconformance requires a capital expense, a change in policy or a procedural change, then management and quality approval is required prior to dispositioning. In this case, check the "management approval" box. Otherwise, disposition the nonconformance as described in item #13 and check the "review" box. Forward the completed NCR to Quality Process Control for review.

#### Please review within 24 hours of receipt.

### Management Review or Approval

- 15. Quality Process Control Engineer instructions: Review the NCR. Evaluate NCR for completeness, accuracy and disposition of nonconformance. Sign, date NCR and forward to originators leader or supervisor. Follow-up as necessary. For requested approval, review and approve or disapprove the disposition. For disapproval, state reasons on NCR and return to NRG or originator. To approve, forward to originators leader or supervisor.
- Originators Immediate Leader or Supervisor: Review the NCR. Review or approve the recommended disposition. To approve. sign, date NCR and forward to originators manager. To disapprove, state reasons on NCR and return to NRG or originator. Forward approved NCR to Originators Manager.
- 17. Originators Manager: Review the NCR, Review or approve/disapprove the recommended disposition. Complete as in item 16. Forward approved NCR to Quality Process Engineer. Attach supporting notes, records, or documents from any resolution process.

### Corrective Action Request and Approval

- 18. For repetitive or significant adverse conditions to quality, request corrective action. Any employee may request corrective action.
- 19. Quality Assurance manager will approve/disapprove request for corrective action and date.
- 20. Quality Process Engineer, Quality Assurance Manager or Quality Auditor will assign a corrective action number.

# APPENDIX 12

# Company-Wide Corrective Action Request and Report

COMPANY-WIDE CORRECTIVE ACTION REQUEST AND REPORT

Complete each item

(See Instructions on Reverse Side)

1. CARR No.   2. Corrective Action Tit	de:	3. Date Opened:
4. Nonconformance, Audit Finding, Prot	olem or Complaint Descri	nion:
Honomormanico, Amini I minig, 1100	nem or complaint Descrip	, a
<u> </u>		
5. CA Requester: 6. Manager/Champio	in Responsible for Implem	nentation: 7. Proposed Implementation Date:
8. Team Members and Telephones		9. QA Manager Approvai:
•		
10. Containment Actions, if any:		
•		
11. Root Cause(s):		
11. Root Cause(s).		
12. Venity Corrective Action(s):		
13. Implementation of Permanent Correct	nve Actions:	
14. Actions to Prevent Recurrence:		
14. Actions to Prevent Recurrence.		
15. Lessons Learned; Who can Benefit is	rom Lessons Learned?	
16. Preparer(s) Name(s)/Date:	17. Approval:	Responsible Manager Name, Signature, Date:
18. Review/Approve: QA Manager		
		mached?

# COMPANY-WIDE CORRECTIVE ACTION REQUEST AND REPORT INSTRUCTIONS (CARR INSTRUCTIONS) (See "Conducting Corrective Actions" SOP for details.)

- Month, day and year this CARR was initiated.
- Name of person requesting corrective action.
- Completed by employee requesting corrective action or Quality Assurance Manager.
- Assigned by Quality Assurance. Format is CARRYearMonthDay-#. Example CARR9300520-01.
- Assigned by Quality Assurance. Examples: 1) Acme Electric Audit Finding 005, 2)
   Employee Concern Lack of Water Fountains or 3) Product Failure, Acme Electric.
- Name of manager responsible for achieving corrective action. This will be determined by Quality Assurance Manager by consensus.
- Name and Telephone Numbers of Corrective Action Team members completed by responsible manager.
- This is an estimate of the date that the corrective action will be fully implemented. Changes require approval by Quality Assurance.
- Quality Assurance Manager reviews items 1 through 8, grants approval to initiate corrective action.
- Specify what was done to verify the effectiveness of the corrective action before it was made permanent.
- 11. Identify the root causes for the problem condition.
- Specify what was done to verify the effectiveness of the corrective action before it was made permanent.
- 13. Specify what corrective action was implemented to solve the problem.
- 14. What actions were taken to prevent recurrence of the problem. Specify.
- From the problem analysis and the corrective action, what lessons were learned and who can benefit from the lessons learned.
- Name of person(s) completing items 10 through 15.
- 17. Approval of the corrective action as implemented by responsible manager.
- Review and approval of corrective action closure by Quality Assurance Manger.

## APPENDIX 13

CARR_

# TRACKING STATUS FOR CORRECTIVE ACTION

	Dates
Request for Corrective Action:	
CA Sent to Responsible Party:	
CA Returned for QA Manager Approval:	
CA Returned to Responsible Party to Begin Action:	

Status Comments		
	·	

Conclusion of Corrective Action Memo to Requester (Optional): _



National Environmental Testing Inc.,

Cambridge Division

Laboratory Quality Assurance Plan



# National Environmental Testing Inc., Cambridge Division Laboratory Quality Assurance Plan

# Prepared By:

NET Cambridge Division 12 Oak Park Bedford, Massachusetts 01730 (617) 275-3535

Approved:	NET Division Manager	Date:
Approved:	NET Director of Analytical	Date:
Approved:	NET QA Coordinator	Date:
Approved:	NET Director of Quality Ass	Date:
Approved:		Date:
Annroved.		Date:

QUALITY ASSURANCE PLAN NET INC., CAMBRIDGE Revision: 2

Revision Date: 10/01/92

## INTRODUCTION

This Laboratory Quality Assurance Plan describes the quality assurance and quality control systems in place at NET Cambridge. The Quality Assurance Program at Cambridge is based on the belief that quality is the key to maintaining leadership in the analytical industry.

### MANAGEMENT POLICY STATEMENT

NET subscribes to the highest standard of quality in its analytical and technical services programs. Specifically, the Company's policies are:

- To conduct laboratory operations within the context of a well defined quality assurance program;
- To communicate the scope and content of the QA Program internally, and to train each employee in its application;
- To report data to customers only when it has met NET's full QA requirements;
- To remove from commercial availability any analysis offered by a NET laboratory if that laboratory fails to demonstrate that it can consistently perform that analysis to NET's high quality standard based on the Interlaboratory Testing Program;
- To resolve to the client's full satisfaction any questions concerning validity or accuracy of analytical data reported by NET to the customer;

NET Cambridge is committed to providing quality services to our clients while complying with state and federal regulatory agencies. Implementation of the policies, principles and practices defined in the Laboratory Quality Assurance Plan is the responsibility of every NET Cambridge employee. This document provides guidance to employees for fulfilling their responsibilities and serves as a statement of the laboratory's commitment to quality to external parties.

# OUALITY ABBURANCE PLAN NET INC., CAMBRIDGE Revision: 2 Revision Date: 10/01/92

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10.	Calibration Procedures and Frequencies	Revision Da 3	te: 10/01/92 10/01/92
	10.1 Laboratory Standards 10.2 Standards Traceability 10.3 GCMS Calibration 10.4 GC Calibration 10.5 ICP Calibration 10.6 Graphite Furnace Calibration 10.7 pH Meter Calibration 10.8 Auto-Analyzer Calibration 10.9 TOC Calibration 10.10 Ion Chromotograph Calibration 10.11 Mercury Analyzer Calibration 10.12 Balance Calibration 10.13 Oven Temperature Check 10.14 Refrigerator Temperature Check	n ,	
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13.	Control Chart Program	3	10/01/92
	13.1 General Policy 13.2 Types of Control Charts 13.3 Analyses Charted 13.4 Generating Control Charts 13.5 Interpreting Control Charts 13.6 Review of Control Charts 13.7 Responding to Out of Control	Conditions	
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	14.1 Immediate Corrective Action 14.2 Long Term Corrective Action 14.3 Documentation of Corrective A	Actions	

·	NET II Revisi	Y ASSURANCE PLAN NC., CAMBRIDG2 ion: 2 ion Date: 10/01/92
15. Data Evaluation and Reporting	3	10/01/92
15.1 Data Reduction 15.2 Data Evaluation 15.3 Data Review Process 15.4 Data Reporting		
16. Audits and Approvals	3	10/01/92
16.1 System Audits 16.2 Performance Audits		
17. QA Reports to Management	3	10/01/92

QUALITY ASSURANCE PLAN

NET INC., CAMBRIDGE Section: 1 Page: 1 of 5

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## 1.0 Laboratory Organization

National Environmental Testing Inc. is a nationwide network of environmental testing laboratories. The Cambridge Division formerly Cambridge Analytical Associates, was acquired by NET in September, 1989.

#### Cambridge Organizational Structure 1.1

Figure 1.1 is the Organizational chart for the Cambridge Division. The responsibilities of key individuals within the Cambridge Division of NET are summarized in section 1.2.

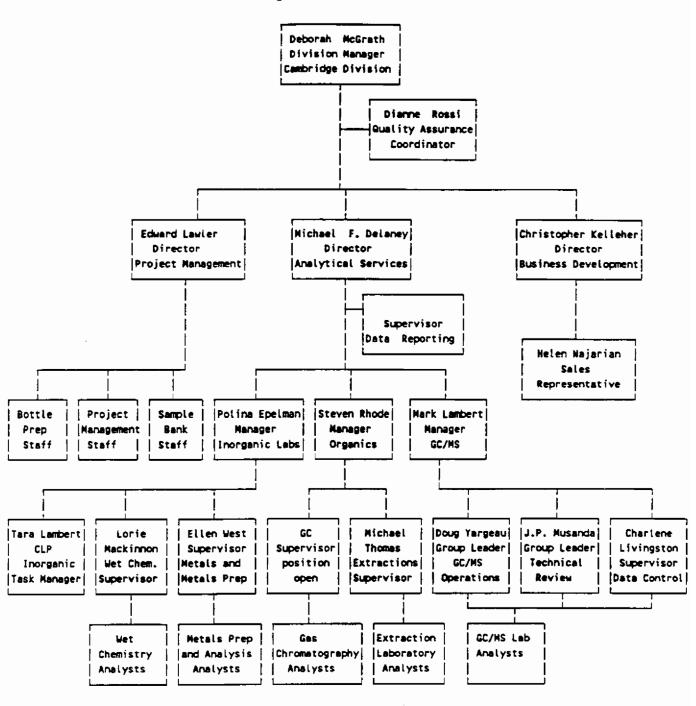
QUALITY ASSURANCE PLAN NET INC., CAMBRIDGE

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Fig. 1.1
NET Cambridge Division
Organizational Chart



QUALITY ASSURANCE PLAN

NET INC., CAMBRIDGE Section: 1 Page: 3 of 5

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#### Description of Responsibilities 1.2

The responsibilities of the individuals associated with this Ouality Assurance/Quality Control (QA/QC) program and illustrated in Figure 4.1 are as follows:

## o <u>Division Manager</u>

The Division Manager oversees technical operations and business development, including marketing, capital budgeting, contract negotiation, management review and quality assurance.

## o Quality Assurance Coordinator

The Quality Assurance Coordinator is responsible development, application, and evaluation of quality control and quality assurance procedures for environmental analytical programs. This responsibility involves:

- 1. monitoring laboratory activities to determine conformance with authorized quality assurance policy, and implementing appropriate steps to ensure adherence to quality assurance programs;
- evaluating data quality and maintaining records on related QC charts and other pertinent information;
- 3. administering intralaboratory and interlaboratory QA efforts;
- reviewing performance evaluation results;
- 5. preparing quality assurance reports to management; and
- 6. managing a corrective action system.

## o Director of Project Management

The Director of Project Management is responsible for client support and service functions, and internal technical support staff. These responsibilities include planning and managing the technical, business development, and human resource functions of the Project Management Department to meet company and program goals.

#### QUALITY ASSURANCE PLAN MET INC., CAMBRIDGE

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#### o The Project Manager

Project Managers are responsible for communication of the work plan and initiating work plan revisions in accordance with the client's directions. This responsibility includes:

- communicating the analytical capabilities of the laboratory accurately;
- ensuring that the expectations and requests of the client are communicated to the laboratory managers;
- reviewing logged in jobs for accuracy and completeness, and sending the client an acknowledgement of receipt;
- informing the client of project progress;

#### o <u>Director of Analytical Services</u>

The Director of Analytical Services is responsible for operation and coordination of the organics and inorganics laboratories. This responsibility includes:

- organizing the personnel, equipment, and materials in the manner required to meet the objectives of the analytical tasks of the project;
- maintaining contact with the project manager in areas of technical concern, and advising on progress, needs, and potential problems of the various tasks.

#### Laboratory Managers

Laboratory Managers for GCMS, Inorganics and Extraction & GC are responsible for directing the activities and monitoring the performance of supervisors and analysts to ensure that they are adhering to high technical standards and to the budget and task schedules. This includes:

- providing technical directions in conduct of laboratory operations and resolving day to day operation's issues; and
- reviewing analytical data for validity, clarity, and compliance with project requirements.

QUALITY ASSURANCE PLAN MET INC., CAMBRIDGE

Section: 1 Page: 5 of 5

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#### o Lab<u>oratory Supervisors</u>

Supervisors of each laboratory are responsible for ensuring that analysts are meeting the production and quality objectives of the project. This responsibility includes:

- training analysts in specified laboratory QC and analytical procedures;
- verifying that laboratory QC and analytical procedures are being followed as specified; and
- 3. reviewing sample QC data at least daily. This will include examination of raw data such as chromatograms (and checking of calculations for a minimum of 10% for the samples analyzed) as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

#### o Laboratory Analysts

Analysts are responsible for each task identified in the scope of work. They are responsible for performing the laboratory technical activities within their tasks. In execution of their duties, the analysts are expected to:

- assist in planning for each phase of their tasks and in defining objectives and activities;
- 2. respond to work plan revisions related to their tasks;
- advise their supervisors of progress, needs, and potential problems of their tasks.

#### o Sample Custodian

Sample Custodians ensure that samples are distributed and returned in accordance with internal chain-of- custody procedures. This includes:

- signing for the incoming field samples and verify the data entered on the chain-of-custody forms;
- entering sample information into a computerized system for tracking and reporting;
- documenting the transfer of samples within the facility.

QUALITY ASSURANCE PLAN NET INC., CAMBRIDGE

Section: 1 Page: 5 of 5

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QUALITY ASSURANCE PLAN MET INC., CAMBRIDGE

Section: 2 Page: 1 of 1

Revision: 3

Revision Date: 10/01/92

#### 2. Training

#### 2.1 Requirements

All analysts are required to demonstrate proficiency in the analyses they will be performing prior to working on actual samples. The training encompasses the analytical procedures to be utilized, the elements of quality control to be associated with the procedure, and the necessary safety information. All of these elements are included in each Standard Operating Procedure. Training is conducted by senior laboratory personnel, and requires that each analyst be familiar with the SOP associated with the task, observe an experienced analyst perform the analysis, work under direct supervision, and finally demonstrate proficiency at the analysis. In addition to this internal training, employees are encouraged to participate in short courses available from instrument manufacturers and professional development seminars.

#### 2.2 Documentation

Each analyst has a personal file that includes a job description, a resume and a training record. All of these are reviewed and updated every six months concurrent with the employees performance planning and review schedule.

QUALITY ABSURANCE PLAN NET INC., CAMBRIDGE Section: 2 Page: 1 of 1

Revision: 3

Revision Date: 10/01/92

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QUALITY ASSURANCE PLAN
NET INC.. CAMBRIDGE

NET INC., CAMBRIDGE Section: 3 Page: 1 of 6

Revision: 3

Revision Date: 10/01/92

#### 3.0 SAMPLE CUSTODY

#### 3.1 Chain-of-Custody Procedures

The purpose of chain-of-custody procedures is to document in a legally defensible manner, the transfer of custody for each sample from collection through analysis to disposal. The sample custody procedures to be used conform to the guidelines of the U.S. EPA Contract Laboratory Program, and are performed under the supervision of the Sample Custodian. The Sample Custodian will have primary responsibility for ensuring chain-of-custody procedures are followed and all documentation is properly executed.

#### 3.2 Sample Receipt

When samples arrive at NET-Cambridge, the Sample Custodian is responsible for signing the air bills, and documenting the condition of the locked or sealed shipping box on the custody form. He/She then checks the temperature of the cooler, and records this temperature in a logbook. Information on the condition of the samples is recorded on the chain of custody form and may also be recorded in a logbook if requested for a project of client. Sample label information (see Figure 3.1) is checked against the custody record, (Figure 3.2). For HAZWRAP and NEESA samples the pH of all preserved samples, except volatiles is checked and recorded. Samples are then logged into the Laboratory Information Management System and are assigned laboratory identification numbers in (LIMS), serialized ascending sequence. The sample log-in record will include the sample number, dates of collection and receipt, the condition of the samples at receipt, the assigned laboratory number, sample preparation, sample distribution and other pertinent information. Sample labels that include the laboratory's internal sample number, the job number which allows samples to be grouped appropriately, the clients name and sample identification, the date of receipt and the disposal date are generated. The sample custodian is responsible for affixing internal sample labels to each sample. laboratories are informed that samples have been received through scheduled generation of scheduling lists.

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The purpose of chain-of-custody procedures is to document in a legally defensible manner, the transfer of custody for each sample from collection through analysis to disposal. The sample custody procedures to be used conform to the guidelines of the U.S. EPA Contract Laboratory Program, and are performed under the supervision of the Sample Custodian. The Sample Custodian will have primary responsibility for ensuring chain-of-custody procedures are followed and all documentation is properly executed.

#### 3.2 Sample Receipt

When samples arrive at NET-Cambridge, the Sample Custodian is responsible for signing the air bills, and documenting the condition of the locked or sealed shipping box on the custody He/She then checks the temperature of the cooler, and form. records this temperature in a logbook. Information on the condition of the samples is recorded on the chain of custody form and may also be recorded in a logbook if requested for a project of client. Sample label information (see Figure 3.1) is checked against the custody record, (Figure 3.2). For HAZWRAP and NEESA samples the pH of all preserved samples, except volatiles is checked and recorded. Samples are then logged into the Laboratory Information Management System (LIMS), and are assigned laboratory identification numbers in serialized ascending sequence. The sample log-in record will include the sample number, dates of collection and receipt, the condition of the samples at receipt, the assigned laboratory number, sample preparation, sample distribution and other pertinent information. Sample labels that include laboratory's internal sample number, the job number which allows samples to be grouped appropriately, the clients name and sample identification, the date of receipt and the disposal date are generated. The sample custodian is responsible for affixing internal sample labels to each sample. The laboratories are informed that samples have been received through scheduled generation of scheduling lists.

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#### 3.3 Sample Storage and Security

The samples are secured in the Sample Bank refrigerator and maintained at approximately 4°C until they are Withdrawn by laboratory analysts. All samples withdrawn from the Sample Bank must be signed for by both the issuing and receiving parties. Figure 3.3 is a copy of the internal Chain-of Custody used for this purpose. Upon Withdrawal, the receiving party is responsible for maintaining the samples in his/her possession or in view at all times when the samples are outside of the storage area. Once the analyst has finished using the samples, they must be returned to the Sample Bank, and the internal Chain-of-Custody is again used to document the transfer.

The NET Cambridge facility has restricted access at all times. All doors are kept locked and guests must be signed in and escorted while in the facility.

#### 3.4 Sample Disposal

All samples are held for a minimum of 60 days after submission of the final report. A list of samples ready to be purged can be generated from the LIM system.

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#### 3.5 Laboratory Document Control

The goal of the Document Control Program is to assure that all documents for a group of samples will be accounted for when the project is completed, and will be maintained in such a way as to facilitate reconstruction of the receipt, preparation, analysis and reporting of each sample. For each sample delivery group, or internal job, a permanent job folder is created to maintain the following information:

- o original chains of custody
- o sample login record
- o copy of client's order confirmation
- o All raw data and results on pre-printed data sheets or copies of laboratory logbooks. All handwritten data entered in ink, and corrections are made with a single line through the error, entering the correct information, and initialing and dating the correction.
- o copy of report sent to client
- documentation of important communication with the client
- o copies of corrective actions and non-conformances associated with the samples

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- o copy of report sent to client
- o documentation of important communication with the client
- o copies of corrective actions and non-conformances associated with the samples

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# Figure 3.1 Example Sample Label

CHEMICAL ANALYSIS:	
SAMPLE DATE:	PRESERVATIVE:
Client	None Sterii
Project/Site	HNO, MeOi
Date/Time	H ₂ SO ₄ Other
Sample ID	ZnAc/NeOH
Location	N₄OH
Calinatas	

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Figure 3.2 Example Chain-of-Custody

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PT 1 - ORIGINAL PT 2 MET Project Manager-Yellow PT 3-Customer Copy-Pink

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# Figure 3.3 Internal Chain-of-Custody Form

N	7						
Ш		la C	:AMB	RIDG	E DI	VISI	ON

Signing samples out of the Sample Bank

Requested by:___

#### INTERNAL CHAIN-OF-CUSTODY FORM

Department (Circle) EX GC IN ME SV VOA

__ Date/Time Needed:_

Date/Time:_

	inquished by							
A Signing samples into the Sample Bank			_					
Relinquished by:	Received by:	Received by:						
B Signing samples into the Sample Bank				Date/Time:				
Relinquished by:				Received by:	Received by:			
C Signing samples into the Sample Bank				Dete/Time:				
Relinquished by:				Received by:				
Sample Number Matrix Pres.	<u></u> f	deturne	<u>d</u>	Depleted	Comments			
(type) (type)	A	В	C	(check)	(test type, etc.)			
1.								
2								
3								
4.								
5.								
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14.	<u> </u>							
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17.								

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#### 4.0 MATERIALS PROCUREMENT AND CONTROL

#### 4.1 Standards Material Procurement

All materials for preparing standards should be of the highest quality available. The only standard certifications currently recognized by the USEPA are referred to as "USEPA Certified" and "A2LA Certified". While use of these materials is not required by USEPA, these materials are of adequately known purity and identity for use in USEPA analytical methods.

Each laboratory group will maintain a listing of acceptable standards manufacturers to be used to guide the ordering of standard materials and for providing information on the sources of materials in use.

When neat, high purity bulk materials are used to prepare standards, they will be obtained in high purity. If the purity is less than 97%, the reason for using this material will be documented as to why a higher purity material could not be obtained. Any standard solution prepared from a neat materials with a purity less than 97% will have its nominal concentration adjusted for the purity of the material.

#### 4.2 Reagent Quality Control

Reagents used in the laboratory are of analytical reagent grade or higher purity. Reagent lots are checked for acceptable purity by the analysis of reagent blanks. NET has arranged for its vendors to set aside reagent lots which have been checked for purity so that subsequent orders can be filled from the already QCed lots. Reagents are dated upon receipt and stored in a designated reagent storage room which is equipped with ventilated solvent cabinets, blow out windows, epoxy flooring, and a diked perimeter. Smaller quantities are stored in ventilated solvent cabinets in the laboratories and in accordance with the MSDS storage requirements.

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#### 5.0 FACILITIES AND EQUIPMENT

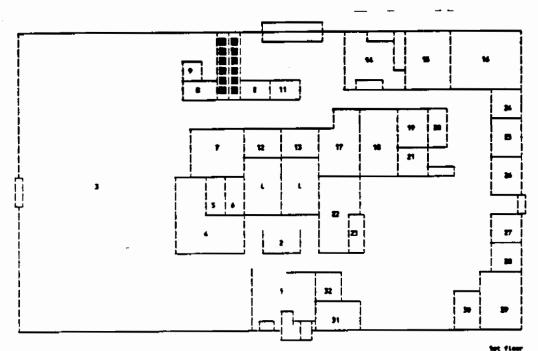
NET continues to invest in new facilities and equipment to support the internal growth of its businesses, to maintain an appropriate level of protection for its staff, and to improve the safety and efficiency of the work environment. trong demand for analytical services requires continual expansion of capacity, and the increasingly stringent quality demands of the industry often require refit of existing laboratories to perform new procedures. The Cambridge Division of NET is situated in a new 40,000 sq.ft. custom designed laboratory. A floor plan is presented as Figure 5.1.

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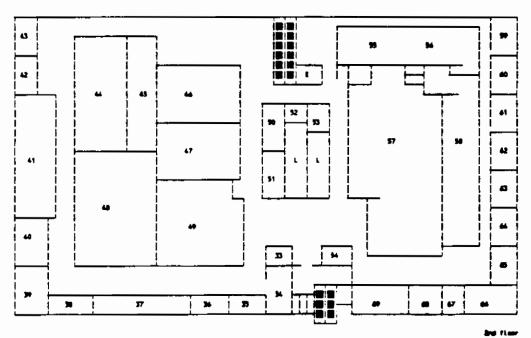
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## Figure 5.1 Cambridge Floor Plan



MET, Combridge Divolat (20,000 sq.ft, int floor) (40,000 sq.ft, total)



MET, Combridge Simples (20,080 eq.ft, 2nd films) (40,080 eq.ft, total)

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## Cambridge Division

	(1st Floor)			(2nd Floor)	
£	Area	# sq. ft.	#	Area	# sq. ft.
1	Reception	420	33	Computer Room	<b>9</b> 5
2	Copy Area	200	34	Conference Room	270
3	Storage	9,000	35	Office	93
4	Accounting	430	36	Office	81
5	Personnel	88	37	Office	308
6	Office	88	38	Office	80
7	Bottle Preparation/Storage	352	39	Office	122
8	Effluent pH Adjustment Equipment	129	40	Office	108
9	Electrical Room	82	41	Office	555
10	Loading Dock	511	42	Office	94
11	Elevator Room	76	43	Office	149
12	Electrical Closet	139	44	Pesticide Laboratory	852
13	Service Closet	108	45	Glassware Cleaning	500
14	Hazardous Waste Storage	243	46	Conventional Chemistry Laboratory	648
15	Solvent Storage	115	47	Metals Preparation Laboratory	648
16	Mechanical Room	675	48	Organics Preparation Laboratory	1,480
17	Log-in/Sample Bank	412	49	Metals Preparation Laboratory	971
18	Walk-in Refrigerator	451	50	Records Storage	200
19	Walk-in Freezer	208	51	Ash Analysis Laboratory	154
20	Storage	139	52	Utilities	69
21	Sampling Equipment	116	53	Storage	93
22	Cafeteria	<b>26</b> 0	54	Copy Center	116
23	Computer Room	193	55	GC Laboratory	<b>60</b> 0
24	Hazardous Waste Coordinator	81	56	Semivolatiles GC/MS Laboratory	634
25	Office	93	57	Volatile Organics Laboratory	2,100
26	Conference Room	105	58	GC/MS Data Processing	1,030
27	Director, Business Development	81	59	Project Manager	116
28	Administrative Assistant	81	60	Project Manager	81
29	Vice President/Division Manager	250	61	Project Manager	104
30	Director of Program Management	104	62	Project Manager	104
31	Office	216	63	Supervisor, GC/MS Operations	104
32	Library	104	64	Office	104
			65	Office	93
L	Lavatory		66	Director, Analytical Services	250
E	Elevator		67	Office	93
			68	Quality Assurance Director	180
			L	Lavatory	
			•	Elevator	

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## 5.2 Major Instrumentation

Table 5.1 lists the major instrumentation used at NET Cambridge

Table 5.1

YEAR MANUFACTURED	INSTRUMENT MODEL	USE	SERIAL NUMBER
986	HP5970-M\$	ABN Analysis	2637A01576
987			2807A10979
	HP5890-GC	ABN Analysis	2631A09163
		•	2750A16248
<b>98</b> 6	HP5970-MS	VOC Analysis	2604A01Z20
u	•	•	2716A10346
	•		2716A10357
987	W		2905A12115
•	•	u	2905A12116
287	HP5890-GC	VOC Analysis	2618A07866
•	e.		2728A12606
<b>m</b>	•		2728A12607
39		#	2921A23814
		•	2921A23815
84	HP5995C GC/MS	VOC Anelysis	2413A00479
P83	HP\$830	VOA Screening	1728A03191
<b>283</b>	NP5890	Pesticides,	2413A06104
	HP5890		2518A05170
	MP5890	<b>#</b>	2518A05226
35	HP5890	•	2750A17912
	HP5890	•	2908A21578
	HP5890		<b>29175</b> P00022
5,1985,	HP5880	Pesticides and	1941A00519
		PHCs	21417A03257
			2303A04978
			2417A06599
91	ABC Labs 1002B GPC	org. extract	8548254
		cleanup	
86	Waters HPLC	Organics	712000744

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Table 5.1

YEAR MANUFACTURED	ENSTRUMENT MODEL	USE	SERIAL NUMBER
989	JY 50 ICP	Metals	609
1991	Jarrelt-Ash AtomScan 1CP	Metals	<b>3</b> 6582
985 , 1986 1986	PE-5000 GFAA	Metals	124347 126282
984	PR-2380 a) Autosampler	Metals	126914
992	PE-	Metals	51002L8123
984	Spectro-Products Hg Analyzer	Hercury	587
987	PE 1310 IR	TPH/OG	133751
987	Dionex QIC Ion Chromatograph	Inorganics	0258

In addition to this major instrumentation, all laboratories are fully edmeters, spectrophotometers, extraction glassware, balances) required for facilities contain appropriate benches, hoods, and safety equipment which Corporate safety managers.

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#### 6.0 INSTRUMENT MAINTENANCE

NET-Cambridge follows a well-defined program to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument downtime. Adequate supplies of spare parts such as GC columns, syringes, septa, injection port liners, and electronic parts are maintained in the laboratories so that they're available when needed.

Preventive maintenance procedures such as lubrication, source cleaning, detector cleaning, and the frequency of such maintenance are performed according to the procedures outlined in the manufacturer's instrument manual. Chromatographic carrier gas purification traps, injector liners, and septa are cleaned or replaced regularly. Precision and accuracy data are examined for trends and excursions beyond established control limits that suggest instrument malfunction. Maintenance must be performed when there is evidence of degradation of peak resolution, shifting of the calibration curves, decreased sensitivity, or failure to meet one or more of the quality control criteria. The preventive maintenance performed on major laboratory instrumentation is summarized in Table 6.1.

Instrument logbooks are maintained by all the laboratories at NET Cambridge. They are used to record instrument use, calibration, maintenance and repairs.

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#### Table 6.1 Routine Maintenance for Major Instrumentation

Instrument	Maintenance Procedure/Schedule	\$	pere Parts
Gas Chromatography/ Mass Spectrometry (GC/MS)	1. Replace pump oils as needed 2. Change septs as needed 3. Change gas line dryers as needed 4. Replace electron multiplier as needed	2. 3.	Syringes Septa Various electromponents Plumbing suppitube fittings
Ges Chrometograph (GC)	<ol> <li>Change septa as needed</li> <li>Change gas line dryers as needed</li> <li>Leak check when installing new analytical column</li> </ol>	2.	Syringes Septa Various elec- components
	<ol> <li>Periodically check inlet system for residue buildup</li> </ol>	4.	Plumbing supp tube fittings
Purge and Trap Sample Concentrator	<ol> <li>Replace trap as needed</li> <li>Decontaminate system as required by blank analysis</li> <li>Leak check system</li> </ol>	2.	Spare traps Electronic components ar circuit board Plumbing supp tube fittings
Graphite Furnace Atomic Absorption Spectrophotometer	<ol> <li>Change graphite contact rings as necessary</li> <li>Change D2 background connection lamp as necessary</li> <li>Clean quartz windows as necessary</li> </ol>	2.	Contact ring: D2 arc lamp
Inductively Coupled Plasma Spectrometer (ICP)	<ol> <li>Clean torch assembly and mixing chamber when discolored or after 8 hours of running high dissolved solids samples</li> <li>ICAPs are under service contracts which provide for quarterly routine maintenance service.</li> </ol>		Spare torch a argon chamber Spare coil

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Table 6.1 (cont'd.)
Maintenance Procedures and Schedule For Major Instrumentation

Instrument	Maintenance Procedure/Schedule	Spere Parts
Spectroproducts Hg Analyzer	<ol> <li>Clean tubing and quartz cell as necessary</li> <li>Clean aspirator as necessary</li> </ol>	1. Quartz cella 2. Aspirator
Technicon Autoanalyzer II	1. Inspect pump tubes after each 8 hour run: replace if discolored or distorted	1. Pump tubes, glass coil, flow cells
pH Analyzer	1. After use in solutions containing free oil and water, wash the electrode in an electrode cleaning solution and water.Rinse thoroughly with water. Immerse the lower third of the electrode in 1+9 NCL for 10 minutes to remove any film formed. Rinse thoroughly with water. Excep electrode properly filled with appropriate filling solution.	solution y er,
Total Organic Carbon Analyzer	<ol> <li>Replace tubing as needed.</li> <li>Replace injector septum after every 100 injections or when leakage is apparent.</li> <li>Prepare fresh reagent daily.</li> <li>Check scrubber daily, repack tube when tin is one-half consumed.</li> </ol>	1. Tubing 2. Septa 3. 20-mesh granular ti

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#### 7.0 OUALITY ASSURANCE OBJECTIVES

Quality control and quality assurance objectives are used to provide analytical data of known, consistent, and defensible quality. Data quality is defined in terms of data quality objectives. These are the qualitative and quantitative statements reflecting the end-user's requirements for precision, accuracy, completeness, representativeness, comparability.

NET Cambridge employs a well defined internal Quality Control (QC) program, a system of specific activities designed and implemented by the laboratory to ensure that a high level of quality is achieved. These include blanks, spikes, duplicates, laboratory control samples, calibration verification standards which are submitted for analysis at regular frequencies, in order to ensure that the end-user's data quality objectives are met.

Analytical environmental work is regulation driven. Therefore, control limits listed in this LQAP are established by EPA methods and protocols or are experimentally determined NET Cambridge criteria. The quality control objectives and the quality control measures and frequencies are outlined in Tables 7.1 and 7.2 respectively.

#### 7.1 Accuracy

Accuracy is a measure of the degree of agreement between an analyzed value and the true or accepted reference value where it is known. Accuracy is usually expressed in terms of error, bias, or percent recovery. Accuracy in the laboratory is assessed by the regular analysis of known standards.

#### 7.2 Precision

Precision is a measure of the mutual agreement among individual measurements of the same parameter under similar conditions. It is an estimate of variability of measurements. Common analytical terms used to describe precision include repeatability, which is associated with a single analyst (within-laboratory) and reproducibility. which is associated with different analysts in different laboratories (between laboratory). Precision within-laboratory measurements is expressed as standard deviation, coefficient of variation, or relative percent difference. Precision in the laboratory is assessed by the regular analysis of duplicate samples.

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#### 7.3 Completeness

Completeness is a measure of the amount of valid data obtained from the analytical measurement system. Completeness is expressed as a percentage of the number of valid measurements that should have been or were planned to be collected.

Ideally, all the expected measurements will be obtained, and all will be valid. However, realistically, some samples may be lost in laboratory accidents or some results may be deemed questionable based on internal QC procedures. Due to the variable nature of the completeness value, the objective will be to have data completeness for all samples received for analysis as high as possible.

#### 7.4 Representativeness

Representativeness is a measure of how closely the measured results reflect the actual concentration or distribution of the chemical compounds in the sample. Sampling will be performed by the client. Sample handling protocols (e.g., storage, preservation and transportation) have developed to preserve the representativeness of been collected samples. Proper documentation will establish that protocols have been followed and that sample identification and integrity have been assured.

#### 7.5 Comparability

Comparability is a measure of how closely sample data generated by NET Cambridge compares with sample data generated by another organization. NET Cambridge will achieve comparability by operating within the instrument linear range and by strict adherence to analytical protocols. The use of published analytical methods, standard reporting units and thorough documentation will ensure meeting this objective. Our participation in EPA and state administered PEs provides a frequent check on the comparability of our data.

#### 7.6 Quality Control Objectives

The quality control objectives associated with specific analyses are described in Table 7.1.

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Table 7.1
Volatile Organic Compounds - CLP 3/90 Protocols

9C Check	Frequency	Analyte/Parameter Checked	Control Limit	:
BFB Tune	once per 12 hour sequence	Relative Ion Abundances	per 3/90 CLP crite	
			specified in OLMO1	.0, D-26/VOA
Holding Blank	one per week	Any project analyte	≤ 5 x CRQL	
Method Blank	one per analytical sequence	2- butanone	≤ 5 x CROL	
	of up to 12 hours	methylene chloride	≤ 5 x CRQL	
		acetone	≤ 5 x CRQL	
		any other project analyte	≤ CRQL	
Cont. Calibration	one per 12 hour sequence	all target compounds	per 3/90 CLP crit	eria specifie
Verification		minimum RF, Maximum % D	in OLM01.2, D-29/	VOA
			Aqueous	<b>Şoils</b>
Surrogate Spike	every sample, blank, and	dq-1,2-dichloroethane	76-114%	70-121%
Recover i es	standard is spiked	dg-toluene	88-110X	84-138%
		4-bromof Luorobenzene	86-115X	59-113%
Matrix Spike	one set per 20 samples of	1,1-dichloroethylene	61-145%	59-172%
Recoveries ^l	similar matrix, per case of	trichloroethylene	71-120%	62-137%
	field samples received,	benzene	76-127%	66-142%
	whichever is more frequent	toluene	<b>76-125%</b>	59-139%
		chlorobenzene	75-130%	60-133%
Precision ¹	one MS/MSD pair per 20	1,1-dichloroethylene	< 14 RPD	< 22 RPD
	samples of similar matrix,	trichloroethylene	≤ 14 RPD	≤ 24 RPD
	or per case of field samples	benzene	≤ 11 RPD	< 21 RPD
	received, whichever is more	toluene	≤ 13 RPD	< 21 RPD
	frequent	chlorobenzene	< 13 RPD	< 21 RPD

RRF- Relative Response Factor

CRQL - Contract Required Detection Limit

RPD- Relative % Difference

 $^{^{1}}$  matrix spike recoveries and precision limits are advisory only - per DLMO1.2 E-20/VOA

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Table 7.1 Volatile Organic Compounds - Modified Method 624/8240

9C Check	Frequency	Analyte/Parameter Checked	Control Limit	
BFB Tune	once per 24 hour sequence	Relative Ion Abundances	per 3/90 CLP crite specified in DLMO1	
Holding Blank	one per week	Any project analyte	≤ 5 x RL	
Method Blank	one per day per instrument	2- butanone methylene chloride acetone any other project analyte	<pre>     5</pre>	
Cont. Calibration Verification	one per 24 hour sequence	all target compounds minimum RF, Maximum % D	per 3/90 CLP crite in OLMO1.2, 0-29/	
			Aqueous	Soils
Surrogate Spike Recoveries	every sample, blank, and standard is spiked	d4-1,2-dichloroethane dg-toluene 4-bromofluorobenzene	76-114% 88-110% 86-115%	70-121% 84-138% 59-113%
Matrix Spike Recoveries	one set per 20 samples of similar matrix	1,1-dichioroethylene trichioroethylene benzene toluene chlorobenzene	61-145% 71-120% 76-127% 76-125% 75-130%	59-172% 62-137% 66-142% 59-139% 60-133%
Precision	one duplicate spike per 20 samples of similar matrix	matrix spike compounds	≤ 25% RPO	

RF- Response Factor

³D- Percent Difference

RPD- Relative % Difference

^{*} surrogate and matrix spike recoveries are based on EPA CLP advisory limits.

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Table 7.1 Volatile Organic Compounds - Modified Method 524.2

QC Check	Frequency	Analyte/Parameter Checked	Control Limit
BFB Tune	once per sequence of up to 24 hours	relative ion abundances	per 3/90 CLP criteria specified in OLMO1.0, D-26/VO
Holding Blank	one per week	524 analytes	<b>≤ Q</b> L
Nethod Blank	one per analytical sequence	524 analytes	<u>&lt;</u> QL
Cont. Calibration Verification	one per day of analysis	CC Compounds	RF 70-130% initial calibration
Surrogate Spike Recoveries	added to each sample, blank, and standard	D4-1,2 Dichlorobenzene-d4 Bromofluorobenzene	80 - 100% 80- 100%
Metrix Spike Recoveries	one MS/MSD per set of 20 samples	1,1-dichloroethylene trichloroethylene benzene toluene chlorobenzene	61-145% 71-120% 76-127% 76-125% 75-130%
Precision	one per 20 samples	matrix spike compounds	≤ 25% RPO

QL - Quantitation Limit

RF - Response Factor

RPD - Relative Percent Difference

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Table 7.1
Extractable Organic Compounds - CLP 3/90 Protocols

QC Check	Frequency	Analyte/Parameter Checked	Control Limit
DFTPP Tune	once per 12 hour sequence	relative ion abundances	per 3/90 CLP criteria specified in OLMO1.0, D-40/SV
Method Blank	one per batch of up to 20 samples	phthalate esters of TCL anelyt all other analytes	es <pre></pre>
Cont. Calibration Verification	one per 24 hour sequence	TCL analytes	min. RF, Max. %RSD, and max. % Dif per OLMO1.0,D-47-48/SV
Surrogate Spike	added to each sample,	d5-nitrobenzene (base/neut.) 2-fluorobiphenyl =	Aqueous <u>\$0ils</u> 35-114% 23-120% 43-116% 30-115%
		p-terphenyl # d-phenol (acid) 2-fluorophenol #	33-141% 18-137% 10-110% 24-113% 21-110% 25-121%
		2,4,6-tribromophenol # 2-Chlorophenol-d4 (advisory) 1,2-Dichlorobenzene-d4 #	10-123% 19-122%
Matrix Spike Recoveries ²	one set per every 20 samples of similar matrix, or per case	1,2,4-trichlorobenzene acenaphthene 2,4-dinitrotoluene	39-98% 38-107% 46-118% 31-137% 24-96% 28-89%
	of samples received, whichever is more frequent	pyrene M-nitroso-di-n-propylamine 1,4-dichlorobenzene	26-127% 35-142% 41-116% 41-126% 36-97% 28-104%
		pentachlorophenol phenol 2-chlorophenol 4-chloro-3-methylphenol	9-103x 17-109x 12-110x 26-90x 27-123x 25-102x 23-97x 26-103x
		4-nîtrophenol	10-80% 11-114%
Precision	every MS/MSD pair	1,2,4-trichlorobenzene acenapthene pyrene	≤28 RPD
		2,4-dinitrotoluene M-nitroso-di-n-propylamine 4-dichlorobenzene	≤38 RPD
		pentachlorophenol phenol 2-chlorophenol	<pre>&lt;50 RPD</pre>
		4-chloro-3-methylphenol 4-nitrophenol	<42 RPD <33 RPD <50 RPD <50 RPD

a maximum of one acidic and one basic surrogate may be out of specified limits matrix spike recoveries and precision limits are advisory only per OLMO1.2, E-26/SV

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Table 7.1 Extractable Organic Compounds - Modified Method 625/8270

QC Check	Frequency	Analyte/Parameter Checked	Control Limit
DFTPP Tune	once per 24 hour sequence	relative ion abundances	per 3/90 CLP criteria specified in OLMO1.0, D-40/SV
Method Blank	one per batch of up	phthalate esters	≤ 5 x QL
	to 20 samples	all other 625 analytes	≤ ar
Cont. Calibration Verification	one per 24 hour sequence		in. RF, Max. %RSD, and max. % Di er OLMO1.0,D-47-48/SV
			Aqueous Soils
Surrogate Spike	edded to each sample,	ණ-nitrobenzene (base/neutral)	
Recoveries 1	blank and standard	2-fluorobiphenyl =	43-116% 30-115%
		p-terphenyl =	33-141% 18-137%
		d-phenol (acidic)	10-110% 24-113%
		2-fluorophenol #	21-110% 25-121%
		2,4,6-tribromophenol =	10-123% 19-122%
		2-Chlorophenol-d4 (advisory)	33-110X 20-130X
		1,2-Dichlorobenzene-d4 *	16-110% 20-130%
Matrix Spike	one set per every	1,2,4-trichlorobenzene	39-98% 38-107%
Recoveries ²	20 samples of similar	acenaph thene	46-118% 31-137%
	matrix '	2,4-dinitrotoluene	24- <del>96</del> % 28-89%
		pyrene	26-127% 35-142%
		N-nitroso-di-n-propylamine	41-116% 41-126%
		1,4-dichlorobenzene	36-97% 28-104%
		pentachlorophenol	9-103% 17-109%
		phenol	12-110% 26-90%
		2-chlorophenol	27-123X 25-102X
		4-chloro-3-methylphenol	23-97% 26-103%
		4-nitrophenol	10-80% 11-114%
Precision	one MSD per every	1,2,4-trichlorobenzene	≤28 RPD ≤23 RPD
	20 samples of similar	acenapthene	<31 RPD <19 RPD
	matrix	pyrene	<31 RPD ≤36 RPD
		2,4-dinitrotoluene	<38 RPD <47 RPD
		N-nitroso-di-n-propylamine	
		4-dichlorobenzene	<28 RPD <27 RPD
		pentachlorophenol	≤50 RPD ≤47 RPD
		phenol	42 RPD 435 RPD
		2-chtorophenot	<40 RPD <50 RPD
		4-chloro-3-methylphenol	<42 RPD <33 RPD
		4-nitrophenol	<50 RPD <50 RPD

a maximum of one acidic and one basic surrogate may be out of specified limits

a matrix spike recoveries and precision limits are advisory only per OLMO1.2, E-26/SV

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Table 7.1 Pesticides and PCBs- CLP 3/90 Protocols

OC Check	Frequency	Analyte/Parameter Checked	Control Limit	t
Linearity	once per calibration sequence	CFs of 3 concentrations of of each single response analytes	% RSO < 20 compounds	(up to two >20 % but < 30%
Continuing Calibration	every 12 hours	all target compounds	≤ 25% RPO	
Verification		Resolution of peaks	100% for PER 90% for ind	Compounds A or B compounds
		Breakdown	DDT and Endr	
Instrument Blank	every 12 hours	all target compounds	< .5 CRQL	
Method Blank	one per 20 samples of similar matrix or per extraction batch	all target compounds	< CRQL	
Surrogate Spike Recoveries	added to each sample blank and standard	Tetrachloro-m-xylene Decachlorobiphenyl	60-150 % red (advisory)	overy
			Aqueous	Soil
Matrix Spike and Duplicate (advisory only)	one per 20 samples of . similar matrix	Lindane Heptachlor Aldrin Dieldrin Endrin 4,4'-DDT	56-123% 40-131% 40-120% 52-126% 56-121% 38-127%	46-127% 35-130% 34-132% 31-134% 42-139% 23-134%
Precision	one MSD per 20 samples of similar matrix	Lindane Heptachlor Aldrin Dieldrin Endrin 4,4'-DDT PCB 1248 *	≤15 RPD ≤20 RPD ≤22 RPD ≤18 RPD ≤21 RPD ≤27 RPD ≤30 RPD	<pre>&lt;50 RP</pre>

CF - Calibration Factor

CRQL - Contract Required Calibration Limit

RSD - Relative Standard Deviation

RPD - Relative Percent Difference

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Table 7.1 Pesticides and PCBs- Modified Method 6082/80803

C Check	Frequency	Analyte/Parameter Checked	Control Limit	
Linearity	once per calibration sequence	CFs of 5 concentrations of of each single response analytes	0.995 correl	ation coefficier
		CFs of 3 concentrations of each PCB of interest	0.995 correl	ation coefficier
Continuing Calibration Verification	every 12 hours	all target compounds	≤ 25% RPD	
Instrument Blank	every 12 hours	all target compounds	< .5 RL	
Method Blank	one per 20 samples of similar matrix or per extraction batch	all target compounds	<u>&lt;</u> RL	
Surrogate Spike Recoveries	added to each sample blank and standard	Tetrachioro-m-xylene Decachiorobiphenyl	60-150 % rec (advisory)	overy
Laboratory Control Sample	one per 20 samples of similar matrix or per extraction batch	single response analytes or PCB 1016 and 1260	limits to be using lab ge	•
			Aqueous	_Soil_
Matrix Spike and Duplicate (advisory only)	one per 20 samples of similar matrix	Lindane Heptachlor Aldrin Dieldrin Endrin 4,4'-DDT PCB 1248*	56-123% 40-131% 40-120% 52-126% 56-121% 38-127% 50-150%	46-127% 35-130% 34-132% 31-134% 42-139% 23-134% 50-150%
Precision	one MSD per 20 samples of similar matrix	Lindane Heptschlor Aldrin Dieldrin Endrin 4,4'-DDT PCB 1248 *	≤15 RPD ≤20 RPO ≤22 RPO ≤18 RPD ≤21 RPD ≤27 RPD ≤30 RPO	≤50 RP ≤31 RPD ≤43 RPD ≤38 RPD ≤45 RPD ≤50 RPD ≤30 RPD

^{* -} performed only for sample sets known to have PCB contamination

CF - Calibration factor

RPD - Relative Percent Difference

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Table 7.1 Herbicides- Modified Method 81503

QC Check	Frequency	Anelyte/Parameter Checked	Control Limit
Method Blank	one per batch of up to 20 samples of similer metrix	2,4-D and Silvex	≤ RL
Initial Calibration	one per day of analysis	2,4-D and Silvex	3 point curve
Continuing Calibration Verification	one per 10 samples	2,4-D and Silvex	70 - 130% expected value
Surrogate Spike	added to each sample, blank and standard	DCPAA	40 - 115X
Laboratory Control Sample	one per extraction batch	2,4-D and \$1lvex	50 - 130%

RL - Reporting Limit

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Table 7.1 Metals Analyses CLP 3/90 Protocols

ac Check	Frequency	Analyte/Parameter Checked	Control Limit
Preparation Blank	one per 20 samples of similar matrix	All metals	< CRDL
Initial Calibration Verification	once per instrument calibration	Hg All others	80-120% 90-110%
Initial Calibration Blank	once per instrument calibration	All metals	< CRDL
Interference Check Sample	once per calibration	ICP metals	80-120X
Matrix Spike	one per 20 samples of similar matrix	All metals	75-125%
Laboratory Control Sample	one per 20 samples	All metals	80-120%
Duplicate Sample	one per 20 samples of similar matrix	All metals	≤ 20% RPD
Continuing Calibration Verification	one per 10 analyses	Hg All others	80-120% 90-110%

CRDL - Contract Required Detection Limit]

RPD - Relative Percent Difference

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Table 7.1 Metals Analyses

QC Check	Frequency	Analyte/Parameter Checked	Control Limit
Preparation Blank	one per 20 samples of similar matrix	All metals	<u>&lt;</u> 5 x RL
Initial Calibration	once per instrument	Hg	80-120%
Verification	calibration	All others	90-110%
Initial Calibration Blank	once per instrument calibration	All metals	≤ 5x IDL
Interference Check Sample	once per calibration	1CP metals	80-120%
Matrix Spike	one per 20 samples	Hex. Chrom.	60-130x
	of similar matrix	All others	75-125% °
Analytical Spike	one per 20 samples of similar matrix	All spiked metals	75-125%
Laboratory Control Sample	one per 20 samples	Ag, Sb	80-120x **
capolatory control sample	of similar matrix	Hex. Chrom.	65 - 125 x **
	2. 2	All others	80-120%
Duplicate Sample	one per 20 samples of similar matrix	All metals	≤ 20% RPD
Continuing Calibration	one per 10 analyses	Hg	80-120%
Verification	,	Ali others	90-110%
Initial Calibration Blank	after each CCV	All metals	≤ 5x IDL

RL - Reporting Limit

The absolute value of the blank must be < RL or < 10 times the lowest sample concentration in the preparation batch.

*Advisory limit - There is no corrective action if outside the limit, it is considered an indication of matrix interference.

RPD - Relative Percent Difference

IDL - Instrument Detection Limit

^{**} Advisory limit

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Table 7.1 Conventional Chemistry Analyses

C Check	Frequency An	mlyte/Parameter Checked	Control Limit
Initial Calibration Curve	one per day of analysis	: All analyses	0.995 Correlation Coefficien
Calibration Blank	one per calibration	Att analyses	≤ 5 x DL
Preparation Slank	one per 20 samples of similar matrix or per batch of < 20 samples	All analyses	≤ 5 x DL
Initial Calibration Verification	one per calibration	All analyses	85-115X
Continuing Calibration Verification	one per 10 analyses	All analyses	85-115%
Matrix Spike Recovery	one per 20 samples of similar matrix or per batch of < 20 samples	All enalyses	75-125X
Duplicate Precision	one per 20 samples of similar matrix or per batch of < 20 samples	All analyses	20% RPD
Laboratory Control Sample Recovery	one per 20 samples of similar matrix or per batch of < 20 samples	TKN and TP All others	75-125% 80-120%

DL - Detection Limit

RPD - Relative Percent Difference

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#### References

- 1 US EPA Contract Laboratory Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, Revision 3/90
- ² US EPA, 1984. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. Appendix A. 40 CFR Part 136. Federal Register, Volume 49, No. 209 1984.
- 3 US EPA, 1986. Test Methods for Evaluating Solid Waste Physical/Chemical Methods. SW-846 Third Edition. Office of Solid Waste, US EPA, Washington DC.
- 4 US EPA, 1990. Method 1311 Toxicity Characteristic Leaching Procedure (TCLP). Appendix II. 40 CFR Parts 261,264,265, 268,271,302. Federal Register, Volume 55, N. 126 6/29/1990
- 5 US EPA, 1986. Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water. EPA/EMSL, Cincinnati, Ohio.
- ⁶ APHA, 1985. Standard Methods for the Examination of Water and Wastewater. Sixteenth Edition. American Public Health Association, Washington DC.
- ⁷ US EPA, 1979. <u>Methods for Chemical Analysis of Water</u> and Wastes. EPA 600/4-79-020 (revised March 1983). EPA/EMSL, Cincinnati, Ohio.
- 8 US EPA, 1988. Contract Laboratory Program Inorganic Analysis for Multi-Media, Multi-Concentration, Statement of Work, Revision 3/90. US Contract Laboratory Program . EPA/CLP, Washington DC.

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#### 8. ANALYTICAL PROCEDURES

The analytical methods routinely employed by NET Cambridge are summarized in Table 8.1. In addition to these analytical methods NET Cambridge is prepared to use current CLP protocols for the analysis of volatiles, semivolatiles, pesticides, PCBs, metals, and cyanide. NET-Cambridge is prepared to use methods other than those listed below should the client request it. If alternate methods are required, QC procedures will be provided as a revision to this QA plan.

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## Table 8.1 Organic Analytical Methods

Constituent	Method Reference	Nethod Description
Volatile Organic Compounds	Modified Method 624/8240 ^{1,2}	Purge and trap, gas chromatography mass spectrometry (GC/MS)
Volatile Organic Compounds	Modified Method 524.2 ⁵	Purge and trap, gas chromatography mass spectrometry (GC/MS)
CLP Volatile Organics	SOW 3/90 ⁶	Purge and trap, GC/MS
Volatile Extractables	Method 504 4	Microextraction GC/ECD enalysis
Semivolatile Organics-Acid/Base/ Neutral Extractables	Modified Method 625/8270 ^{1,2}	Two phase extraction, gas chromato- graphy mass spectrometry analysis
CLP Semivolatile Organics	sow 3/90 ⁶	Single phase solvent extraction
Pesticides/PCBs	Modified Method 608/8080 ^{1,2}	Solvent extraction, gas chromato- graphy/ electron capture
CLP Pesticides	sow 3/90 ⁶	Solvent extraction, ECD analysis
Pesticides	505 ⁴ 508 ⁴	Microextraction/ GC/ECD analysis Solvent extraction/ GC/ECD analysis
Herbicides (Aqueous)	Modified Method 615/509B/8150 5,2	Solvent extraction, derivatization, gas chromatography/electron capture detection (GC/ECD)
TCLP/ZHE Extraction	Method 1311 ²	Acidic leaching procedure
Petroleum Hydrocarbons, Total	Modified Methods 418.1	Solvent extraction, IR spectrometry
Petroleum Hydrocarbon Fingerprinting		Solvent extraction, gas chromatography/flame ionization detection (GC/FID)
Oil and Grease	Method 413.1 ^{3,5} Method 413.2 ³	Solvent extraction, gravimetric determination Solvent extraction, infrared spectrophotometry (IR)

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## Organics Methods References

- ¹US EPA, 1984. <u>Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater</u>. Appendix A. 40CFR Part 136. Federal Register, Volume 49, No. 209, 1984.
- ²US EPA, 1986. <u>Test Methods for Evaluating Solid Waste-Physical/Chemical Methods</u>. <u>SW-846 Third Edition</u>. Office of Solid Waste, US EPA, Washington, DC.
- ³US EPA, 1979. <u>Methods for Chemical Analysis of Water and Wastes</u>. EPA 600/4-79-020 (Revised, March 1983). EPA/EMSL, Cincinnati, Ohio.
- US EPA, 1986. Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water. EPA/EMSL, Cincinnati, Ohio.
- ⁵APHA, 1985. <u>Standard Methods for the Examination of Water and Wastewater</u>. Sixteenth Edition. American Public Health Association, Washington, DC.
- OS EPA, 1988. Organic Analysis for Multi-Media, Multi-Concentration, Statement of Work, Revision 3/90.
  US Contract Laboratory Program. EPA/CLP, Washington, DC.

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## Table 8.2 Summary of Inorganic Methods

Constituent	Method Reference	Method Description_	
Sample Digestion	Method 3010/3020/3050 ²	Acid Digestion	
EP Toxicity	Method 1320 ²	Toxicity Extraction	
TCLP Extraction	Method 1311 ²	Acidic Leaching Procedure	
Aluminum	Method 200.7/6010 ^{1,2}	Inductively Coupled Argon Emission Spectroscopy (ICP)	
Antimony	Method 200.7/6010 1,2	ICP	
•	Method 204.2/7041	Graphite Furnace Atomic	
		Absorption Spectrophotometry	
		(GFAAS)	
Arsenic	Method 200.7/6010 1,2	ICP	
	Method 206.2/7060	GFAAS	
	Method 206.3	Hydride generation	
Barium	Hethod 200.7/6010 1.2	ICP	
Beryllium	Method 200.7/6010 1,2	ICP	
Cadmium	Method 200.7/6010 1,2	1CP	
	Hethod 213.2/7131 12	GFAAS	
Calcium	Method 200.7/6010 1.2	I CP	
Chromium	Method 200.7/6010 1,2	1CP	
Cobalt	Method 200.//outu	ICP	
Copper	Method 200.7/6010 1.2	1CP	
Iron	Method 200.7/6010 1,2	ICP	
Lead	Method 200.7/0010	ICP	
_	METHOD 537.5/1421	GFAAS	
Nagnesium	METHOD SOUTH TO	1CP	
Manganèsè	Method 200.7/6010	ICP	
Mercury	Method 245.1/7470 1,2 Method 200.7/6010 1,2	Cold-Vapor AA Spectroscopy	
Not ybdenum	Method 200.7/6010 1,2	ICP	
Nickel	Method 200.7/6010 1,2	ICP	
Potassium Selenium	Method 200.7/6010 1,2	1CP	
Setenium	Method 270.2/7740	GFAAS	
Silver	Method 200.7/6010 1,2	ICP	
21 fAEL	Method 272.2	GFAAS	
\$odium	Method 200.7/6010 1.2	ICP	
Socium Thallium	Method 200.7/6010 1,2	ICP	
(Hetta)	Hethod 279.2/7841 1,2	GFAAS	
Vanadium	Method 200.7/6010 1.2	ICP	
Zinc	Method 200.7/6010 1,2	ICP	
CLP Metals	sow 3/90 4	1CP/GFAAS	
CLY MCTO(2	30 3/ / G	• • • • • • • • • • • • • • • • • • • •	

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# Table 8.2 (cont.) Inorganic Analytical Methods

Constituent	Method Reference	Method Description
Alkalinity	Method 310.1 ¹ Method 350.1 ¹	Potentriometric Titration
Almonia	Method 351.2 1	Colorimetric Sami-automated
	Method 351.2	Slock Digester
Siochem. Oxygen Demend		_
(BOO)	Method 405.1 1	5 days at 20°C
Carbonaceous BOD	Method 405.1 ¹ inhibited	5 days at 20 ⁰ C; nitrogen
Chemical Oxygen Demand (COD)	Method 410.4 ¹	Colorimetric
Chloride	Method 300.1/325.2 1	Ion chromatography; titrimetric
Color	Method 110.2 1	Colorimetric, Pt-Co
Conductivity	Method 120.1 1	Specific conductance
Cyanide, Total	Method 335.2/.3 ^{1,2}	Distiliation, colorimetric
	Method 9012	autometed
Cyanide, Amenable	Method 335.1/9012 ^{1,2}	Chlorination, distillation colorimetric
Cyanide, CLP	sou 3/90 ⁴	Distillation, colorimetric
fluoride	Method 300.1/340.2 1	Ion chromatography, ion selective electrode
Hardness	Method 2340B 3	ICP, by calculation
Hexavalent Cr	Mod.Method 218.5/7195 1,2	Co-precipitation, ICP
Nitrate	Method 300.0/353.2 1	Ion chromatography, automated colorimetric
Nitrite	Method 300.0/353.2 ¹	Ion chromatography,
Orthophosphate	Method 365.3 ¹	automated colorimetric Manual two reagent
рН	Hethod 150.1/9040 ^{1,2}	Potentriometric
Phenolics	Method 420.2 ¹	Colorimetric, automated
Phosphorus, Total	Method 365.3 ¹	Colorimetric, ascorbic acid
Silica	Method 200.7 ¹	Filtration, ICP

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## Table 8.2 (cont.) Inorganic Analytical Methods

Analysis	Method Reference	Method Description
<u>Solids</u> Dissolved (TDS)	Method 160.1 ¹	Gravimetric, dry at 180 C
Suspended (TSS)	Method 160.2 1	Gravimetric, dried at 103-105oC
Total (TS)	Method 160.3 1	Gravimetric, dried at
Volatile (TVS)	Method 160.4 1	103-105 <u>o</u> C Gravimetric, ignition 550 <u>o</u> C
Settlemble (SS)	Method 160.5 1	Volumetric, Imhoff Cone
Sulfide	Method 376.1/9030 1,2	Titrimetric, fodine
Sulfate	Method 300.0 1 Method 375.4/9038 1,2	ion chrometography, Turbidimetric
Total Organic Carbon	Hethod 415.2/9060 1,2	Persulfate oxidation, UV
Total Kjeldahl Nitrogen Turbidity	Method 351.2 1 Method 180.1	promoted Semi-automated colorimetric Nephelometric

## Inorganic Methods References

¹US EPA, 1979. <u>Methods for Chemical Analysis of Water and</u> Wastes. EPA 600/4-79-020 (Revised, March 1983). EPA/EMSL, Cincinnati, Ohio.

²US EPA, 1986. <u>Test Methods for Evaluating Solid Waste-Physical/Chemical Methods</u>. SW-846 Third Edition. Office of Solid Waste, US EPA, Washington, DC.

³APHA, 1985. Standard Methods for the Examination of Water and Wastewater. Sixteenth Edition. American Public Health Association, Washington, DC.

⁴US EPA, 1988. Contract Laboratory Program Inorganic Analysis for Multi-Media, Multi-Concentration, Statement of Work, Revision 3/90. US Contract Laboratory Program. EPA/CLP, Washington, DC.

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## 9.0 Detection and Reporting Limits

## 9.1 Instrument Detection Limits

The concentration equivalent of the smallest signal which is distinguishable from background instrument noise. The determination of IDL's is done for metals quarterly per CLP protocols. The IDL is 3 times the average of 3 standard deviations each independently determined on non-consecutive days by 7 replicate analyses of a spiked reagent water standard under normal instrument operating conditions

#### 9.2 Method Detection Limits

The Method Detection Limits is defined as the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero. This concentration is determined through multiple analyses (including all sample preparation steps) of a sample of a given matrix. The standard deviation of the results of these analyses is multiplied by 3, and the resulting value is considered the method detection limit. This procedure for determining the MDL is from 40 CFR Part 136 Appendix B Federal Register Vol. 49, No. 209 - 10/26/84). MDLs are done annually.

#### 9.3 Reporting Limits

The reporting limit is the concentration down to which the laboratory can confidently report quantitative data. Reporting limits must always be greater or equal to the MDL or IDL. The judgement of the analysts, and the needs of the client are considered when determining reporting limits. The reporting limits for specific methods and matrices are listed in Table 9.1.

#### 9.4 Contract Required Quantitation Limits

The CRQLs are the reporting limits required for EPA CLP analyses. These are defined in the CLP Statements of Work, and are listed in Table 9.1 for each CLP analysis performed.

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Table 9.1
Analytes and Reporting Limits

			CLP SON 3/90	
		CROL	CROL	CROL
		Water	Low Soil	Medium Soil
VOLATILES	CAS No.	US/L	ug/Kg	ug/Kg
1. Chloromethane	74-87-3	10	10	1200
2. Bromomethane	74-83-9	10	10	1200
3. Vinyl Chloride	75-01-4	10	10	1200
4. Chloroethane	75-00-3	10	10	1200
5. Hethylene Chloride	75-09-2	10	10	1200
6. Acetone	67-64-1	10	10	1200
7. Carbon Disulfide	75-15-0	10	10	1200
8. 1,1-Dichloroethene	75-35-4	10	10	1200
9. 1,1-Dichloroethane	75-34-3	10	10	1200
0. 1,2-Dichloroethene (total)	540-59-0	10	10	1200
1. Chloroform	67-66-3	10	10	1200
2. 1,2-Dichloroethane	107-06-2	10	10	1200
3. 2-Butanone	78-93-3	10	10	1200
. 1,1,1-Trichloroethane	71-55-6	10	10	1200
5. Carbon Tetrachloride	56-23-5	10	10	1200
6. Bromodichloromethane	75-27-4	10	10	1200
7. 1,2-Dichloropropane	78-87-5	10	10	1200
8. cis-1,3-Dichloropropene	10061-01-5	10	10	1200
9. Trichloroethene	79-01-6	10	10	1200
O. Dibromochloromethane	124-48-1	10	10	1200
1. 1,1,2-Trichloroethane	79-00-5	10	10	1200
2. Benzene	71-43-2	10	10	1200
3. trans-1,3-Dichloropropene	10061-02-6	10	10	1200
4. Bromoform	75-25-2	10	10	1200
5. 4-Methyl-2-pentanone	108-10-1	10	10	1200
6. 2-Hexanone	591-78-6	10	10	1200
7. Tetrachloroethene	127-18-4	10	10	1200
3. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200
9. Toluene	108-88-3	10	10	1200
0. Chlorobenzene	108-90-7	10	10	1200
1. Ethyl Benzene	100-41-4	10	10	1200
2. Styr <del>ene</del>	100-42-5	10	10	1200
3. Xylenes (total)	1330-20-7	10	<u>10</u>	1200

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Table 9.1 Analytes and Reporting Limits (cont'd.)

		Modified Method 624 ² /8240 ³	Modified Method 8240 ³	TCLP Method 1311 Modified 8240 ³
		QL	QL	QL
VOLATILES	CAS No.	ua/L	ua/Ka	ua/L
1. Acetone	76- <del>64</del> -1	5	5	•
2. Benzene	71-43-2	5	5	25
3. Bromodichloromethane	75-27-4	5	5	•
4. Bramoform	75-25-2	5	5	-
5. Bromomethane	74-83-9	5	5	-
6. 2-Butanone	78-93-3	5	5	100
7. Carbon disutfide	75-15-0	5	5	•
8. Carbon Tetrachloride	56-23-5	5	5	25
9. Chlorobenzene	108-90-7	5	5	25
D. Chloroethane	75-00-3	5	5	•
o. Chtoroethane	13-00-5	,	•	
1. 2-Chloroethylvinyl ether	100-75-8	5	5	•
2. Chloroform	67-66-3	5	5	25
3. Chloromethane	74-87-3	5	5	•
4. Dibromochloromethane	124-48-1	5	5	-
5. 1,2-Dichlorobenzene	<b>9</b> 5-50-1	5	5	-
5. 1,3-Dichlorobenzene	541-73-1	5	5	-
7. 1,4-Dichlorobenzene	106-46-7	5	5	•
8. 1,1-Dichloroethane	75-34-3	5	5	•
2. 1,2-Dichloroethane	107-06-2	5	5	25
0. 1,1-Dichloroethene	75-35-4	5	5	25
1. trans 1,2-Dichloroethene	540-59-0	5	5	•
2. 1,2-Dichloropropane	78-87-5	5	5	•
3. cis-1,3-Dichloropropene	10061-01-5	5	5	•
4. trans-1,3-Dichloropropene	10061-02-6	5	5	-
5. Ethyl Benzene	100-41-4	5	5	•
•	E01.70.7		•	
5. 2-hexanone	591-78-6	5	5	•
7. 4-methyl-2-pentanone	108-10-1 75-09-2	5 · 5	5 5	_
B. Methylene Chloride	100-42-5	5	5	
7. styrene	79-34-5	5	5	-
0. 1,1,2,2-Tetrachloroethane	17-34-7	,	,	-
1. Tetrachloroethene	127-18-4	5	5	25
2. Toluene	108-88-3	5	5	-
3. 1,1,1-Trichloroethane	71-55-6	5	5	•
4. 1,1,2-Trichloroethane	79-00-5	5	5	-
5. Trichloroethene	79-01-6	5	5	25

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Table 9.1
Analytes and Reporting Limits (cont'd.)

		Modified Method 624 ² /8240 ³	Modified Method 8240 ³	TCLP Method 1311 Modified 8240 ³
		QL .	QL	QL
VOLATILES	CAS No.	ug/L	ug/Kg	ug/L
36. Trichlorfluoromethane	75-69-4	5	5	-
57. Vinyl acetate		5	5	-
S8. Vinyl Chloride	75-01-4	5	5	100
59. m-xylene	108-38-3	5	5	-
0. o-xylene	95-47-6	5	5	•
41. p-xylene	106-42-3	5	5	

^{*} HSL extended analysis required

#### QL - quantitation limit

Quantitation Limits listed for soil/sediment are based on wet weight. Those calculated by the laboratory are on a dry weight basis, and will be higher.

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Table 9.1 Analytes and Reporting Limits (cont'd.)

-		Method	
		524 ⁵	
		<del>ο</del> Γ	
		<b>U</b> ater	
OLATILES	CAS No.	ug/L	
. Benzene	71-43-2	0.2	
. Bromobenzene	108-86-1	0.4	
. Bromochloromethane	75-01-4	0.4	
. Bromodichloromethane	75-27-4	0.4	
. Bromoform	75-25-2	0.3	
. Bromomethane	74-83-9	0.3	
. n-Butylbenzene	104-51-8	0.4	
. sec-Butyibenzene	135-98-8	0.2	
. tert-Butylbenzene	98-06-6	0.3	
O. Carbon Tetrachloride	56-23-5	0.6	
1. Chlorobenzene	108-90-7	0.2	
2. Chloroethane	75-00-3	0.4	
3. Chloroform	67-66-3	0.7	
4. Chloromethane	74-87-3	0.5	
5, 2-Chlorotolu <del>ene</del>	95-49-8	0.4	
6. 4-Chlorotoluene	106-43-4	0.3	
7. 1,2-Dibromo-3-Chloropropane	<del>96</del> -12-18	0.8	
8. Dibromochloromethane	124-48-1	0.3	
9. 1,2-Dibromoethane	106-93-4	0.3	
O. Dibromomethane	74-95-3	0.3	
1. 1,2-Dichtorobenzene	95-50-1	0.4	
2. 1,3 Dichtorobenzene	541 <i>-7</i> 3-1	0.2	
3. 1,4 Dichtorobenzene	106-46-7	0.5	
4. Dichlorodifluoromethane	75-71-8	1.5	
5. 1,1-Dichloroethane	75-34-3	0.3	
6. 1,2-Dichloroethane	107-06-2	0.7	
7. 1,1-Dichloroethene	75-35-4	1.3	
8. cis-1,2-Dichloroethene	156-59-4	0.4	
9. trans-1,2-Dichloroethene	156-60-5	0.2	
0. 1,2-Dichloropropane	78-87-5	0.3	
1. 1,3-Dichloropropene	142-28-9	0.3	
2. 2,2-Dichloropropane	590-20-7	0.6	
3. 1,1-dichloropropene	563-58-6	0.3	
4. cis-1,3-Dichloropropene	10061-01-5	0.4	
5. trans-1,3-Dichloropropene	10061-02-6	0.4	

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Table 9.1 Analytes and Reporting Limits (cont'd.)

		Method	
		524 ⁵	
		<b>Q</b> L	
		Water	
VOLATILES	CAS No.	ue/L	
36. Ethyl Benzene	100-41-4	0.3	
37. Hexachlorobutadiene	87-68-3	0.6	
38. Isopropyl benzene	98-82-8	0.4	
39. 4-Isopropyltoluene	99-87-6	0.4	
40. Methylene Chloride	75-09-2	1.7	
41. Hapthalene	91-20-3	2.8	
42. n-Propylbenzene	103-65-1	0.4	
43. styrene	100-42-5	0.4	
44. 1,1,1,2-Tetrachloroethane	630-20-6	0.5	
45. 1,1,2,2-Tetrachioroethane	79-34-5	0.3	
46. Tetrachloroethene	127-18-4	0.4	
47. Toluene	108-88-3	0.2	
48. 1,2,3-Trichlorobenzene	87-61-6	0.9	
49. 1,2,4-Trichlorobenzene	120-82-1	0.7	
50. 1,1,1-Trichloroethane	71-55-6	0.5	
51. 1,1,2-Trichloroethane	79-00-5	0.2	
52. Trichloroethene	79-01-6	0.3	
53. Trichlorfluoromethane	75-69-4	0.6	
54. 1,2,4-Trimethylbenzene	95-63-6	0.4	
55. 1,3,5-Trimethylbenzene	108-67-8	0.4	
56. 1,2,3-Trichloropropane	96-18-4	0.6	
57. Vinyl Chloride	75-01-4	0.4	
58. m-Xylene	108-38-3	0.7	
59. o-Xylene	95-47-6	0.4	
60. p-Xylene	106-42-3	0.7	

QL - Quantitation Limit

Specific detection limits are highly matrix dependent. The Quantitation Limits listed provided for guidance and may not always be achievable.

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## Table 9.1 Analytes and Reporting Limits (cont'd.)

VOLATILES	CAS No.	ue/L	
1. 1,2-Dibromoethane (EDB)	106-93-4	1	
2. 1,2-Dibromo-3-Chloropropene (DBCP)	96-12-8	2	

#### QL - Quantitation Limit

Specific detection limits are highly matrix dependent. The Quantitation Limits listed provided for guidance and may not always be achievable.

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Table 9.1
Analytes and Reporting Limits (cont'd.)

			CLP SON 3/901		
			CRQL	CROL	CROL
			Water	Low Soil	Medium Soi
SEM	IVOLATILES	CAS No.	ug/L	ug/Kg	ug/Kg
1	Phenol	108-95-2	10	330	10.000
-	bis(2-chloroethyl)ether	111-44-4	10	330	10,000
	2-Chlorophenol	95-57-8	10	330	10,000
	1,3-Dichlorobenzene	541-73-1	10	330	10,000
	1,4-Dichtorobenzene	106-46-7	10	330	10,000
٦.	1,4°D (Citto) openzene	100-40-7		330	10,000
6.	1,2-Dichlorobenzene	95-50-1	10	330	10,000
	2-Methylphenol	95-48-7	10	330	10,000
8.	2,2'-oxybis 1-Chloropropane	108-60-1	10	330	10,000
9.	4-Methylphenol	106-44-5	10	330	10,000
10.	N-nitroso-di-n-	621-64-7	10	330	10,000
	dipropylamine				
11.	Hexachloroethane	67-72-1	10	330	10,000
	Nitrobenzene	98-95-3	10	330	10,000
	Isophorone	78-59-1	10	330	10,000
	2-Nitrophenol	88-75-5	10	330	10,000
	2,4-Dimethylphenol	105-67-9	10	330	10,000
		****	10	330	10,000
	bis(2-chioroethoxy)methane	111-91-1		330	10,000
	2,4-Dichlorophenol	120-83-2	10 16	330	10,000
	1,2,4-Trichlorobenzene	120-82-1 91-20-3	10	330 330	10,000
	Naphthalene			330 330	-
20.	4-Chloroaniline	106-47-8	10	350	10,000
21.	Hexach Lorobutadiene	87-68-3	10	330	10,000
22.	4-Chloro-3-methylphenol	<del>59-</del> 50-7	10	<b>33</b> 0	10,000
23.	2-Methylnaphthalene	91-57-6	10	330	10,000
24.	Hexachlorocyclopentadien	77-47-4	10	330	10,000
25.	2,4,6-Trichlorophenol	88-06-2	10	330	10,000
26.	2,4,5-Trichtorophenol	95-95-4	25	800	25,000
	2-Chloronaphthalene	91-58-7	10	330	10,000
	2-Nitroaniline	88-74-4	25	800	25,000
	Dimethylphthalate	131-11-3	10	330	10,000
	Acenaphthalene	208-96-8	10	330	10,000
••	2,6-Dinitrotoluene	606-20-2	10	330	10,000
	3-Nitroaniline	99-09-2	25	800	25,000
	•	83-32-9	10	330	10,000
	Acenaphthene		25	800	25,000
	2,4-Dinitrophenol 4-Nitrophenol	51-28-5 100-02-7	25	800_	25,000

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Table 9.1
Analytes and Reporting Limits (cont'd.)

			CLP SOW 3/901	
		CROL	CRQL	CROL
		Water	Low Soil	Medium Soi
SEMIVOLATILES	CAS No.	Ug/L_	ug/Kg	ug/Kg
36. Dibenzofuran	132-64-9	10	330	10,000
37. 2.4-Dinitrotoluene	121-14-2	10	330	10,000
38. Diethylphthalate	84-66-2	10	330	10,000
39. 4-Chlorophenyl-	7005-72-3	10	330	10,000
phenylether				•
40. Fluorene	86-73-7	10	330	10,000
41. 4-Nitrosniline	100-01-6	25	<b>80</b> 0	25,000
42. 4,6-Dinitro-2-	534-52-1	25	800	25,000
methylphenol				
43. H-nitrosodiphenylamine	6-30-6	10	330	10,000
44. 4-Bromophenyl-	101-55-3	10	330	10,000
phenylether				•
45. Hexachlorobenzene	118-74-1	10	330	10,000
46. Pentachlorophenol	87-86-5	25	800	25,000
47. Phenanthrene	85-01-8	10	330	10,000
48. Anthracene	120-12-7	10	330	10,000
49. Carbazole	86-74-8	10	<b>3</b> 30	10,000
50. Di-n-butylphthalate	84-74-2	10	330	10,000
51. fluoranthene	206-44-0	10	330	10,000
52. Pyrene	129-00-0	10	330	10,000
53. Butylbenzylphthalate	85-68-7	10	330	10,000
54. 3,3'-Dichlorobenzidine	91-94-1	10	<b>33</b> 0	10,000
55. Benzo(a)anthracene	56-55-3	10	330	10,000
56. Chrysene	218-01-9	10	330	10,000
57. bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10,000
58. Di-n-octylphthalate	117-84-0	10	330	10,000
59. Benzo(b)fluoranthene	205-99-2	10	330	10,000
60. Benzo(k)fluorathene	207-08-9	10	330	10,000
61. Benzo(a)pyrene	50-32-8	10	330	10,000
62. Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10,000
63. Dibenz(a,h)anthrace	53-70-3	10	330	10,000
64. Benzo(g,h,i)perylene	191-24-2	10	330	10,000

Specific Quantitation Limits are highly matrix dependent. The Quantitation Limits disted herein are provided for guidance and may not always be achievable.

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Table 9.1 Analytes and Reporting Limits (cont'd.)

		Modified Method 625 ² /8270 ³	Modified Method 8270 ³	TCLP Method 1311 Modified 8270 ³	
		QL	<b>Q</b> L	<b>Q</b> L	
		Water	Soil	Leachate	
SEMIVOLATILES	CAS No.	ug/L	ug/Kg	us/L	
1. Acenephthene	83-32-9	2	40		
2. Acenaphthylene	208-96-8	2	40	•	
3. Anthracene	120-12-7	2	40		
4. Benzo (a) anthracene	56-55-3	2	40	•	
5. Benzo(a)pyrene	50-32-8	2	40	•	
6. Benzo(b)fluoranthene	205-99-2	2	40		
7. Senzo(g,h,i)perylene	191-24-2	2	40		
8. Benzo(k)fluorathene	207-08-9	2	40	•	
9. Benzoic Acid *	65-85-0	2	40	•	
10. Benzyl Alcohol *	100-51-6	2	40	•	
11. 4-Bromophenyl-phenylether	101-55-3	2	40	•	
12. Butylbenzylphthalate	85-68-7	2	40	•	
13. 4-Chloro-3-methylphenol	59-50-7	2	40	20	
14 (para-chloro-meta-cresol)					
15. 4-Chloroaniline *	106-47-8	2	40	•	
16. bis(2-chioroethoxy)methane	111-91-1	2	40	•	
17. bis(2-chloroethyl)ether	111-44-4	2	40		
18. bis(2-chloroisopropyl)ether	108-60-1	2	40	+■	
19. 2-Chloronaphthalene	91-58-7	2	40	·•	
20. 2-Chiorophenol	95-57-8	2	40	•	
21. 4-Chlorophenylphenylether	7005-72-3	2	40		
22. Chrysene	218-01-9	2	40	1.	
23. Di-n-butylphthalate	84-74-2	2	40	•	
24. Di-n-octylphthalate	117-84-0	2	40		
25. Dibenz(a,h)anthracene	53-70-3	2	40	•	

QL - Quantitation Limit

Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

Quantitation limits for soil/sediments are based on wet weight. The quantitation limits calculated by the laboratory are on a dry weight basis and will be higher.

^{* -} HSL compound extended analysis required

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Table 9.1
Analytes and Reporting Limits (cont'd.)

<del></del> -		Modified Method 625 ² /8270 ³	Modified Method 8270 ³	TCLP Method 1311 Modified 8270 ³	
		OL	<u>e</u> L	QL	
		Water	Soil	Leachate	
BEMIVOLATILES	CAS No.	ug/L	Ug/Kq	U9/L	
26. Dibenzofuran	132-64-9	2	40	-	
27. 1,2-Dichlorobenzene	95-50-1	2	40	•	
28. 1,3-Dichlorobenzene	541-73-1	2	40	•	
29. 1,4-Dichlorobenzene	106-46-7	2	40	20	
30. 3,3'-Dichlorobenzidine	91-94-1	2	40	-	
31. 2,4-Dichlorophenol	120-83-2	2	40	•	
32. Diethylphthalate	84-66-2	2	40	•	
53. Dimethyl phthalate	131-11-3	2	40	•	
34, 2,4-Dimethylphenol	105-67-9	2	40	-	
35. 4,6-Dinitro-2-methylphenol	534-52-1	2	40	•	
36. 2,4-Dinitrophenol	51-28-5	2	40	-	
37. 2,4-Dinitrotoluene	121-14-2	2	40	20	
38. 2,6-Dinitrotoluene	606-20-2	2	40	•	
39. bis-2 Ethylhexyl phthalate	117-81-7	2	40	•	
10. Fluorene	86-73-7	2	40	-	
41. Fluoranthene	206-44-0	2	40	-	
42. Hexachtorobenzene	118-74-1	2	40	20	
43. Hexachlorobutadiene	87-68-3	2	40	20	
44. Hexachlorocyclopentadiene	77-47-4	2	40	•	
45. Mexachtoroethane	67-72-1	2	40	20	
66. Indeno(1,2,3-cd)pyrene	193-39-5	2	40	-	
47. Isophorone	78-59-1	2	40	-	
48. 2-Methylnapthalene *	91-57-6	2	40		
49. 2-Methylphenol *	95-48-7	2	40	•	
50. 4-Methylphenol *	106-44-5	2	40	•	

QL - Quantitation Limit

Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

Quantitation limits for soil/sediments are based on wet weight. The quantitation limits calculated by the laboratory are on a dry weight basis and will be higher.

^{* -} MSL compound extended analysis required

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Table 9.1
Analytes and Reporting Limits (cont'd.)

		Modified Method 625 ² /8270 ³	Modified Method 8270 ³	TCLP Method 1311 Modified 8270 ³	
		QL	QL	QL	
		Water	<b>S</b> oi l	Leachate	
SEMIVOLATILES	CAS No.	ug/L	<b>∪g/</b> Kg	ue/L	
51. N-nitroso-di-n dipropylamine	621-64-7	2	40		
52. N-nitrosodimethylamine	62-75-9	2	40	•	
53. N-nitrosodiphenylamine	6-30-6	2	40		
54. Naphthalene	91-20-3	2	40		
55. 2-Nitroanaline *	88-74-4	2	40	•	
56. 3-Nitroanaline *	99-09-2	2	40	-	
57. 4-Mitroenaline	100-01-6	2	40	•	
58. Nitrobenzene	98-95-3	2	40	20	
59. 2-Nitrophenol	88-75-5	2	40	-	
60. 4-Nitrophenol	100-02-7	2	40	•	
61. Pentachlorophenol	87-86-5	2	40	20	
62. Phenanthrene	85-01-8	2	40	•	
63. Phenol	108-95-2	2	40	-	
64. Pyrene	129-00-0	2	40	•	
65. Pyridine	110-86-1	-	-	20	
66. 1,2,4-Trichtorobenzene	120-82-1	2	40	-	
67. 2,4,5- Trichlorophenol *	95-95-4	2	40	20	
68. 2,4,6-Trichlorophenol	88-06-2	2	40	20	

QL - Quantitation Limit

Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

Quantitation limits for soil/sediments are based on wet weight. The quantitation limits calculated by the laboratory are on a dry weight basis and will be higher.

^{* -} MSL compound extended analysis required

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Table 9.1 Analytes and Reporting Limits (cont'd.)

ECT!				CLP SOW 3/901
ECT I			CRGL	CRQL
SCT I			Water	Soil
ESIL	IDES/PCBs	CAS No.	Ug/L	ug/Kg
1.	alpha-BHC	319-84-6	0.05	1.7
	beta-BHC	319-86-8	0.05	1.7
	delta-BHC	319-86-8	0.05	1.7
	gamma-BHC (lindane)	58-89-9	0.05	1.7
	Heptachlor	76-44-8	0.05	1.7
	****	*** ** *		
	Aldrin	309-00-2	0.05	1.7
	Heptachlor epoxide	1024-57-3	0.05	1.7
	Endosulfan I	959-98-8	0.05	1.7
	Dieldrin	60-57-1	0.10	3.3
10.	4,4'DDE	72-55- <del>9</del>	0.10	3.3
11.	Endrin	72-20-8	0.10	3.3
12.	Endosulfan II	33213-65-9	0.10	3.3
13.	4,4'DDD	72-54-8	0.10	3.3
14.	Endosulfan Sulfate	1031-07-8	0.10	3.3
15.	4,4'-DDT	50-29-3	0.10	3.3
16.	Methoxychlor	72-43-5	0.50	17.0
	Endrin ketone	53494-70-5	0.10	3.3
18.	Endrin aldehyde	7421-36-3	0.10	3.3
19.	alpha-Chlordane	5103-71-9	0.05	1.7
20.	gamma - Chil ordane	5103-74-2	0.05	1.7
21.	Toxaphene	8001-35-2	5.0	170.0
	Aroctor-1016	12674-11-2	1.0	33.0
	Aroctor-1221	11104-28-2	2.0	67.0
	Aroclor-1232	11141-16-5	1.0	33.0
	Aroctor-1242	53469-21-9	1.0	33.0
		4		
	Aroclor-1248	12672-29-6	1.0	33.0
•	Aroctor-1254 Aroctor-1260	11097-69-1 11096-82-5	1.0 1.0	33.0 33.0

There is no differentiation in the preparation of low and medium soil samples in this method for the analysis of pesticides and aroctors.

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Table 9.1
Analytes and Reporting Limits (cont'd.)

			Modified Method 608 ² /8080 ³	Modified Method 8080 ³	TCLP Method 1311 ⁴ Modified 8080 ³
			QL	QL	<b>G</b> L
_			Vater	\$oil	Leachate
F	PESTICIDES/PCBs	CAS No.	ue/L	ug/Kg	ug/t
1.	alpha-BHC	319-84-6	0.10	4.0	-
2.	beta-BHC	319-86-8	0.10	4.0	-
3.	delta-BHC	319-86-8	0.10	4.0	•
4.	gamma-BHC (lindane)	58-89-9	0.10	4.0	2.0
5.	Heptachior	76-44-8	0.10	4.0	4.0
6.	Aldrin	309-00-2	0.10	4.0	•
7.	Weptachlor epoxide	1024-57-3	0.10	4.0	4.0
8.	Endosulfan I	959-98-8	0.10	4.0	-
9.	Dieldrin	60-57-1	0.10	4.0	-
10.	4,4'DDE	72-55-9	0.10	4.0	-
11.	Endrin	72-20-8	0.10	4.0	2.0
12.	Endosulfan II	33213-65-9	0.10	4.0	-
13.	4,4'DDD	72-54-8	0.10	4.0	•
14.	Endosulfan Sulfate	1031-07-8	0.10	4.0	-
15.	4,4'-DDT	50-29-3	0.10	4.0	•
16.	Methoxychlor	72-43-5	0.5	4.0	20
17.	Endrin aldehyde	7421-36-3	0.10	4.0	•
18.	Chlordane	57-74-9	1.0	200	20
19.	Toxaphene	8001-35-2	1.0	70	20
20.	Aroctor-1016	12674-11-2	1.0	70	•
21.	Aroclor-1221	11104-28-2	··· 1.0	70	-
22.	Aroctor-1232	11141-16-5	1.0	70	•
23.	Aroctor-1242	53469-21-9	1.0	70	-
24.	Aroctor-1248	12672-29-6	1.0	70	•
25.	Aroclor-1254	11097-69-1	1.0	70	-
26.	Aroclor-1260	11096-82-5	1.0	70	

Specific Quantitation Limits are highly matrix dependent. The Quantitation Limits listed herein are provided for guidance and may not always be achievable.

Quantitation Limits listed for soil/sediment are based on wet weight. The Quantitation Limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis, will be higher.

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Table 9.1 Analytes and Reporting Limits (cont'd.)

		Modified Method 509b ⁶ /8150 ³	Modified Hethod 8150 ³	TCLP Method 1311 Modified 8150 ³
		QL	<u>e</u> L	<b>QL</b>
		Water	\$oil	Leachate
MERBICIDES	CAS No.	ue/L	ug/Kg	uq/L
1. 2,4-D	94-75-7	0.1	1	20
2. Silvex	93-72-1	0.1	1	2.0
ISCELLANEOUS ANALYS	ES	QL	<b>QL</b>	
1. PMCs "fingerpri	nting"	(ug/L)	(ug/g)	
gasoline		100	2	
mineral spiri	ts	100	2	
kerosene		100	2	
#2 Fuel Oil/D	iesel	100	2	
#4 Fuel Oil		100	2	
#6 Fuel Oil		100	2	
Lubricating 0	il	100	2	
coal tar		100	2	
2 TPHs (418.1)		2.0 mg/L	100 mg/Kg	(20 g sample)
3. Oil and Grease			QL	
Infra-red ( M	ethod 413.2 ⁷ )	2 mg/L	100 mg/Kg	
gravimetric (	Modified Method	413.1') 5 mg/L	250 mg/Kg	
4. PCBs in Oil		1 mg/Kg		

QL - Quantitation Limit

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Table 9.1
Analytes and Reporting Limits (cont'd.)

		CLP SOL	∤ 3/90 ⁸
		CRDL	CRDL
	Analyte	ug/t	<b>Ug/</b> 9
1.	Aluminum	200	40
2.	Antimony	60	12
3.	Arsenic	10	2
4.	Barium	200	40
5.	Beryllium	5	1
6.	Çadınium	5	1
7.	Calcium	5000	1000
8.	Chromium	10	2
9.	Cobait	50	10
10.	Copper	25	5
11.	1ron	100	20
12.	Lead	3	.6
13.	Magnes i um	5000	1000
14.	Manganese	15	3
15.	Mercury (HGCVA)	0.2	.04
16.	Nickel	40	8
17.	Potassium	5000	1000
18.	Selenium	5	1
19.	Silver	10	2
20.	Sodium	5000	1000
21.	Thallium	10	2
22.	Vanadium	50	10
23.	Zinc	20	4

The CLP CRDL are the instrument detection limits obtained in pure water. The detection limits for samples may be considerably higher depending on the sample matrix.

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Table 9.1 Analytes and Reporting Limits (cont'd.)

		TCLP Method 1311 ⁴ Method 6010 ³	Methods	Analysis 200.7 ⁷ /6010 ³	7000 s	
		QL	IDL		10	
		mg/L	ug/L	mg/kg	ug/L	mg/kg
1.	Atuminum	-	50	10		-
2.	Antimony	•	30	6.D	6.0	1.2
3.	Arsenic	0.5	100	20	2.0	0.40
4.	Barium	10.0	10	2.0	•	-
5.	Beryllium	•	1.0	0.20	•	-
6.	Boron	•	100	20	-	-
7.	Cadmium	0.1	3.0	0.60	1.0	0.20
8.	Calcium	•	100	20	-	-
9.	Chromium	0.5	5.0	1.0	2.0	0.40
10.	Cobalt	-	5.0	1.0	•	-
11.	Copper	•	5.0	1.0	1.0	0.20
12.	Hex. Chromium	•	50	0.50	-	-
13.	1 ron	•	10	2.0	-	-
14.	Lead	0.5	100	200	2.0	0.40
15.	Magnes i um	-	100	20	-	-
16.	Manganese	-	5.0	1.0	-	-
17.	Mercury	0.01	0.2*	0.10*	-	-
18.	<b>Mot ybdenum</b>	•	100	20	-	•
19.	Nickel	•	5.0	1.0	•	•
20.	Potassium	•	500	100	-	-
21.	Selenium	0.2	100	20	1.0	0.20
22.	Silver	0.1	5.0	1.0	1.0	0.20
23.	Sodium	•	100	20	-	•
24.	Thallium	-	100	20	2.0	0.40
25.	Tin	-	50	10	10	2.0
26.	Vanedi um	•	5.0	1.0	-	•
27.	Zinc	•	5.0	1.0	-	•

Instrument detection limits are updated quarterly.

^{*} Mercury analysis by Methods 245.1⁷/7470³

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Table 9.1 Analytes and Reporting Limits (cont'd.)

	Quantitation Limits			
Analyte	Aqueous		\$oft	
1. Acidity	20	<b>mg</b> /L		
2. Alkalinity	2.0	mg/L		-
3. Ammonia	0.10	mg/L	1	mg/Kg
4. Biochemical Oxygen Demand (BCD)	4.0	mg/L		-
5. Biochemical Oxygen Demand, Carbonaceous (CBOD)	6.0	mg/L		-
6. Chemical Oxygen Demand (CCC)	10	■g/L		-
7. Chloride	0.10	mg/L	1	mg/Kg
8. Color	5.0	color units		•
9. Cyanide, Total	0.010	mg/L	0.5	mg/Kg
O. Cyanide, Amenable	0.010	mg/L	0.5	mg/Kg
1. Fluoride	0.10	mg/L	0.5	mg/Kg
2. Hardness	1.0	mg/L		-
3. Hydrogen Ion (pH)	0.20	pH units		-
4. Kjeldahl Nitrogen, Total (TKN)	0.5	mg/L	25	mg/Kg
5. Witrate	0.10	mg/L	1	mg/Kg
6. Witrate/Witrite	0.020	mg/L	0.2	mg/Kg
7. Organic Carbon, Total (TOC)	0.10	mg/L		-
8. Orthophosphate	0.010	mg/L	0.1	mg/Kg
9. Phenols	0.010	mg/L	0.2	mg/Kg
0. Phosphorus, Total	0.010	mg/L	1	mg/Kg
1. Residue, Total (TS)	5.0	mg/L		•
2. Residue, Non-Filterable (TSS)	5.0	mg/L		-
3. Residue, Settleable	0.10	mg/L/hour		•
4. Residue, Volatile (TVS)	5-0	mg/L		•
5. Residue, Filterable (TDS)	5.0	mg/L		-
6. Specific Conductance	5.0	umho/cm2		-
7. Sulfate	0.5	mg/L	4	mg/Kg
8. Sulfide	1.0	mg/L	10	mg/Kg
9. Sulfite	5.0	mg/L		-
0. Turbidity	0.50	units		-

Limits of detection may vary with sample matrices.

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#### 10.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures and frequency for the instrumentation which will be used in the determination of the parameters of interest.

### 10.1 Laboratory Standards

Materials used for calibration, internal standards and surrogate standards will be of the highest purity available and will be obtained through the U.S. Environmental Protection Agency Pesticide and Industrial Chemicals Repository or a suitable commercial source. The calibration procedures outlined here are those routinely used in the laboratory. The frequency of calibration is also included in Table 7.1.

## 10.2 Standards Traceability

All materials, whether high purity bulk materials or prepared solutions, will have the following information, at a minimum, recorded into an analytical standards logbook: identity, supplier, catalog number, production lot number, date received, reported purity or concentration, and expiration date. This information will be recorded when the material is received, or no later than the first time the material is used.

All analytical standards and spiking solutions will have a unique identification consisting of a name, number and the preparation or received date. This identification will be clearly recorded on the label of any bottle containing this material. By consistently using this identification, the material can be traced back to the original source material.

Documentation of all standard preparations will be recorded in logbooks. The volume and numerical reference of all analytical standards or spiking solutions used in the preparation of another standard will be recorded in the standard preparation logbook.

All calibration standards must be verified against and independently prepared standard from a second manufacturer, or a different lot from the same manufacturer.

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## 10.3 GCMS Calibration Procedure

full calibration procedure outlined below is performed initially and then again whenever the criteria for the continuing calibration check are not met. The tuning procedure is done every 12 hours for as long as the calibration is valid, and prior to an initial calibration.

- At the beginning of each shift that volatile organics analyses are performed, the GC/MS system must be checked to verify that acceptable performance criteria are obtained for bromofluorobenzene (BFB). The performance test must be passed before analyzing any samples, blanks or standards. A tune with decafluorotriphenyl phosphine (DFTPP) is used prior on instruments dedicated to semivolatile analyses.
- Analyze a five point initial calibration sequence using 2. standards prepared following EPA protocols. Calibration check compounds (CCCs) criteria must be met before sample analysis may begin.
- 3. Analyze a continuing calibration check standard prepared following EPA procedures. If the CCC and System Performance Check Compounds (SPCC) do not meet the criteria stated in the CLP SOW 3/90 (OLMO1.2), the source of the problem must be identified and corrected before sample analysis can begin. If these criteria cannot be met, the instrument must have a new initial calibration performed following any necessary maintenance.
- 4. Record all values for the initial and subsequent calibration verifications.

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## 10.4 Gas Chromatograph Calibration Procedures

A full initial calibration procedure is done when continuing calibration criteria are not met or when any major change in instrument hardware or instrument parameters is made.

- Analyze a Resolution Check Mixture standard to ensure that the following criteria is met: The depth of the valley between two adjacent peaks must be greater than or equal to 60% of the height of the shorter peak.
- 2. Analyze a Performance Evaluation Mixture to check endrin and DDT breakdown which must not exceed 20% for either compound or 30% for combined breakdown:
- 3. Analyze Arochlor and toxaphene standards. One concentration of each is analyzed and the retention time and calibration factor for each is determined using a set of 3-5 major peaks.
- 4. Analyze 3 concentrations of Individual Standard Mixtures (5 concentrations for SW846 analyses) containing all of the target list pesticides. The linearity of each compound is determined by calculating the %RSD of the calibration factors. The RSD for each compound should not exceed 20%. (Up to two compounds per column can exceed 20%, but must be less than 30% RSD.)
- 5. Analyze an Instrument Blank. The concentration of any target analyte in this blank must be less than .5 X CRQL.
- 6. Analyze a Performance Evaluation Mixture. The calculated concentrations of each analyte in this mixture should be within 75-125 % of the expected value. The breakdown products, retention time window drift, and peak resolution are also checked. Peaks in the Performance Evaluation Mixture must be 100% resolved. Retention times must fall within the windows specified in the CLP SOW for each compound using the mean retention time from each of the Individual Mixes as the center of each window.
- 7. Samples are run once all of the above standards are run and the acceptance criteria are met.
- 8. No later than 12 hours after the injection of the Instrument Blank, another Instrument Blank followed by the medium level of each of the Individual Standard Mixtures must be run to assess the validity of the continuing calibration. The blank results must be as specified in step #6 above, and the individual standard mixture results must be within 25% of the expected value for each analyte. Resolution between peaks must be > 90%.

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9. If the continuing calibration check (step 9 above) meets the acceptance criteria specified sample analysis may continue for another 12 hours (from the time of the Instrument Blank injection) before repeating the continuing calibration check this time using a Performance Evaluation Mixture rather than the Individual Standard Mixtures.

10. Continue analyzing samples and checking the continuing calibration alternately using both Individual mixtures and the Performance Evaluation Mixture

#### 10.5 ICP Calibration Procedure

- 1. Calibrate the instrument using a blank and one concentration of each element.
- 2. After the calibration standards are run, verify the initial calibration for each element of interest using an EPA Quality Control concentrate or other independent standard at a concentration within the calibration range. If the reported values for the calibration check sample exceed the control limits of 90-110% expected value, the analysis is terminated and corrective action taken.
- 3. Once the system is satisfactorily calibrated, verify the low end of the calibration with an initial calibration blank. The result of this blank analysis must be  $\pm$  5x the IDL for for each element of interest for commercial work or  $\pm$  CRDL for CLP analyses.
- 4. To assure calibration accuracy throughout each analysis run, the calibration check standard must be analyzed after each set of 10 analyses. The calibration check sample is also analyzed after the last analytical sample. If the calibration check sample is out of the control limits of 90-110% expected value, the analysis must be terminated and the instrument recalibrated. All samples analyzed since the last in control calibration check must be reanalyzed.
- 5. To assure low end stability, a blank must be run after each continuing calibration verification sample. The result of the blank analysis must be <u>+</u> the detection limit of each element of interest.
- 6. All values for the initial and subsequent calibrations are reported on a computer generated print out of the analytical sequence.

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## 10.6 Graphite Furnace Calibration Procedure

1. Calibrate the instrument beginning with a blank and three standards, working toward the highest standard.

- 2. After the system is calibrated, verify the initial calibration for each element of interest using an EPA Quality Control concentrate or other independent source at a concentration within the calibration range. The result of the ICV should be within 10% of the expected value. If it is not, the analysis must be terminated and corrective action taken.
- 3. Once the system is satisfactorily calibrated, verify the low end of the calibration with an initial calibration blank. The result if this blank analysis must be ± 5% the IDL for each element of interest for commercial work and ± CRDL for CLP analyses.
- 4. To assure calibration accuracy throughout each analysis run, the calibration check standard must be analyzed at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The calibration check sample is also analyzed after the last analytical sample. If the calibration check sample is outside the control limits of 90-100% expected value, the analysis must be terminated and the instrument recalibrated. All samples analyzed since the last time the calibration check was in control must be reanalyzed.
- 5. To assure low end stability, a blank must be run after each continuing calibration verification sample. The result of the blank analysis must be ± the detection limit of each element of interest.
- 6. All calibration quality control sample results will be printed on the computer generated analysis run information.

#### 10.7 pH Meter Calibration

- 1. Perform an initial operating check of the electrode and meter according to manufacturer's instructions.
- 2. Calibrate the meter according to the operating instructions. Use the standards prepared above.
- 3. Analyze a quality control standard to verify instrument calibration.
- 4. Record all values for initial and subsequent calibration verifications.

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### 10.8 Auto-Analyser

- 1. Calibrate the instrument using the blank and the standards.
- 2. After the instrument is calibrated verify and document the initial calibration using an EPA quality control concentrate or other independent standard at a concentration other than that used for calibration but within the calibration range.
- 3. If the reported values for the calibration check sample exceed the control limits, the analysis is terminated and the problem corrected.
- 4. To assure calibration accuracy throughout each analysis sequence, the calibration check standard must be analyzed at a frequency of 10%. The calibration check sample is also analyzed after the last sample in the sequence. If the calibration check sample is outside the control limits, the analysis must be terminated and the instrument recalibrated. All samples analyzed since the last time the calibration check was in control must be reanalyzed.

## 10.9 Total Organic Carbon Analyzer Calibration Procedure

- 1. Calibrate the instrument according to the procedures in the manufacturer's operating manual.
- 2. Use these working standards at the start of each analysis day to verify that the instrument is functioning properly.
- 3. Analyze an EPA quality control concentrate or other appropriate laboratory control sample. If the results are within the established control limits, the analysis may proceed.
- 4. Document all standard preparations and instrument operating parameters.

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## 10.10 Ion Chromatograph Calibration Procedures

- 1. Calibrate the instrument beginning with the blank and working toward the highest standard.
- 2. After the system is calibrated, verify and document the initial calibration for each analyte of interest using an EPA Quality Control concentrate or other independent standard at a concentration other than that used for calibration but within the calibration range.
- If the reported values for the calibration check sample exceed the control limits, the analysis is terminated and the problem corrected.
- 4. To assure calibration accuracy throughout each analysis run, the calibration check sample must be analyzed at the frequency specified in Table 5.3. The calibration check sample must also be analyzed after the last analytical sample. If the calibration check sample is outside the control limits, the analysis must be terminated and the instrument recalibrated. All samples analyzed since the last time the calibration check sample was in control must be reanalyzed.
- 5. Record all values for the initial and subsequent calibration verifications.

#### 10.11. Mercury Analyzer Calibration Procedure

- 1. Calibrate the instrument beginning with the blank and working toward the highest standard.
- 2. After the system is calibrated, verify and document the initial calibration using an EPA Quality Control concentrate or other independent standard at a concentration other than that used for calibration but within the calibration range.
- 3. If the reported values for the calibration check sample exceed the control limits, the analysis is terminated and the problem corrected.
- 4. To assure calibration accuracy throughout each analysis run, the calibration check must be analyzed at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The calibration check sample is also analyzed after the last sample. If the calibration check sample is outside the control limits, the analysis must be terminated and the instrument recalibrated. All samples analyzed since the last time the calibration check was inside the control limit must be reanalyzed.

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## 10.12 Analytical Balances Calibration

Each analytical balance is calibrated each day of use by weighing at least 3 weights in the appropriate the range of intended use. and certifying that the balance is capable of accurately determining the weight to one decimal point beyond what the intended use requires. The weight sets maintained in the laboratories are checked periodically against a set of Class S certified weights maintained by the QA Coordinator. Daily calibration information is kept in a logbook for each balance, and QA maintains records of its weights inspections. In addition, all analytical balances are under manufacturer service contracts and are inspected by a certified service representative yearly.

### 10.13 Oven Temperature Check

Two separate types of ovens are maintained for the analysis of samples. Each oven is vented and has an exhaust fume hood. One is used for low temperature (<200°C) desiccation and evaporation. This oven has a thermoelectric potentiometer to maintain operating temperature. The temperature is monitored daily with a laboratory grade mercury thermometer and that information is recorded in a logbook.

The other oven is a muffle furnace used for volatile solids and ash determinations. This furnace operates at high temperatures (>200°C) +2°C. The oven temperature is monitored on each day of use and the information is recorded in a log book. Thermometers are calibrated against precision thermometers certified by the National Bureau of Standards. All calibration information is recorded and each thermometer is given a unique ID.

#### 10.14 Refrigerator Temperature Check

Refrigerators are kept at a constant 40 C as measured by a calibrated thermometer. The temperature is monitored at least once daily and this information is recorded in a refrigerator specific log book. Thermometers used to monitor refrigerator temperatures are immersed in water to ensure readings that are representative of sample and extract temperature conditions.

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#### 11.0 GLASSWARE WASHING

Glassware from each group must be separately maintained in order to avoid contamination. There are designated work areas in the dishwashing room for inorganics and organics glassware, and each glassware technician is assigned to only one department at a time.

In addition,

- Glassware containing soil from samples should be rinsed into a waste container -- not down the drain.
- Glassware which has been moistened by organic solvents should be allowed to air-dry in a hood before being rinsed.

#### 11.1. Inorganic Glassware

All volumetrics, graduated cylinders, pipettes, beakers, glass and plasticware used in the trace metal or priority pollutant analyses are cleaned by the following procedures:

- Remove visible dirt by washing with tap water 1.
- Wash glassware/plastic ware with tap water and glassware soap using scrub brushes. The automatic dishwasher can also be used if available. If the dishwasher is used two rinse cycles are necessary to remove all soap.
- Rinse hand washed items thoroughly with tap water.
- 4. Alternately acid and DI rinse in the following order:

1:1 HNO3 DI water 1:1 HCl DI water

This should be done in a sink with an acid trap.

- All beakers and watchglasses must be acid refluxed on a hot plate for one hour using 10 ml of concentrated HNO3. This is done in the laboratory hood by the glassware technician.
- Rinse all glass/plastic ware (including the beakers watchglasses that have been acid refluxed) three times with deionized water.
- Allow glass/plastic ware to dry in clean hood. 7.
- Store clean plasticware in plastic bags.

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## 11.2 Organic Glassware

- Rinse the glassware at least twice with water prior to washing. Use brush to loosen any visible dirt from surface.
- Wash fragile and large pieces with soap and water in the dishroom sink using scrub brushes.
- Any item that is not too fragile or large can be washed in the automatic dishwasher. Make sure that all items are placed securely and that no items will bump into each other. NOTE: Do not put small plastic items in the dishwasher. This will cause jamming and possible breakage of the machine.
- Add 1/3 cup of granular soap to the dishwasher and allow it to run full cycle.
- All glassware, handwashed and dishwasher washed, must be methanol rinsed. This is done by the glassware technician in the fume hood. The waste is collected for disposal in the appropriate waste solvent drum.
- Glassware is allowed to dry in the dish room hood and then is returned to the laboratory drawers.
- Prior to initiating any procedures, the laboratory analyst must rinse all glassware with methylene chloride at least three times.
- If another solvent is going to be used in the glassware, an additional rinse with that solvent should be done after the methylene chloride rinses.

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#### 12.0 QUALITY CONTROL SAMPLES

Several different types of QC samples are used to document the validity of the generated data. Quality control objectives and QC frequencies are outlined in Table 7.1. The following are definitions of the various types of QC samples routinely incorporated into analyses.

### 12.1 Blank Samples

- a. <u>Field Blank</u> (Equipment Blank, Rinsate Blank) A sample of laboratory deionized water which proceeds through all the sample collection equipment after the equipment has been decontaminated. Field blanks should be collected once during a sampling event.
- b. Trip Blank A sample of laboratory water which is placed in an appropriate sample container, handled in the same manner as field samples, and returned to the laboratory with the samples, to assess the possible contamination introduced in transport.
- c. <u>Holding Blank</u> A sample of laboratory water or solid matrix which is placed in an appropriate sample container and stored along with the samples in the refrigerator or storage container to asses any contamination which may be introduced in storage.
- d. <u>Method Blanks</u> A sample of laboratory water or suitable solid matrix that is carried through the entire analytical procedure (digested or extracted, and analyzed). These blanks are handled using the same reagents, surrogates, etc. as the samples in order to assess possible contamination during the analytical process.
- e. <u>Calibration Blanks</u> A sample of laboratory DI water or solvent containing the same reagents at the same concentration as the calibration standards. This blank is used to "zero" the instrument.

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### 12.2 Spiked Samples

a. <u>Surrogate Spike</u> (SS) - Compounds which are added to every blank, sample, matrix spike, matrix spike duplicate and standard in order to evaluate the analytical efficiency of the method in individual sample matrices. The surrogate compounds are chemically similar to the target compounds.

- b. Analytical Spike (AS) An aliquot of digested sample into which a known amount of compound is added. The analytical spike is analyzed immediately and the recovery is calculated in order to assess the matrix effect on the analytical system.
- c. <u>Blank Spike</u> Also called an LCS, this is a volume of laboratory deionized water or clean solid matrix into which a known amount of compound is added. The blank spike is subjected to the entire analytical procedure, and the percent recovery of the spiked compounds is calculated in order to assess the efficiency of the extraction and analysis.
- d. <u>Matrix Spike</u> (MS) (Digestion Spike) An aliquot of sample (water, soil, or sludge) into which a known amount of compounds are added. The matrix spike is subjected to the entire extraction and analytical procedure. The percent recovery of the spiked compounds is calculated in order to assess the appropriateness of the method for that matrix.
- e. <u>Matrix Spike Duplicate</u> (MSD) A second aliquot of the same sample as the the matrix spike that also has a known amount of compound added and is taken through the entire procedure. The percent recoveries of the spiked compounds for both the matrix spike and the matrix spike duplicate are compared in order to assess the precision of the method for that matrix.

### 12.3 Initial Calibration Verification Standard (ICV)

An independent reference standard made from a source different than that of the calibration standards, which is run after each calibration of an instrument to verify that the instrument and standards are operating properly.

### 12.4 Continuing Calibration Verification Standard (CCV)

An analytical standard which is run at a specified frequency (e.g. every 10 samples, or every 2 hours, etc.) to verify the calibration of the analytical system.

### 12.5 Duplicate Samples

A second aliquot of a sample which is carried through sample preparation and analysis procedures to verify the precision of the analytical method for that matrix.

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### 12.6 Internal Standards (IS)

Compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOA), and sample extract (for semi-VOA) at a known concentration prior to the analysis. The internal standards are used as the basis for quantitation of the target compounds.

### 12.7 Interference Check Standard (ICS)

An standard used in ICAP analysis that contains low concentrations of analytes of interest, and high concentrations of interfering elements. The ICS is run at the beginning and at the end of an analysis sequence to ensure that the procedures used to eliminate interfering responses are operating properly.

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### 13. CONTROL CHART PROGRAM

control charts are an important and useful part of a QA/QC program. They are also regarded as a significant tool of a total quality management (TQM) program. In addition to containing a large amount of information of the performance of a process over time, the visual aspect of a control chart allows this information to be expediently understood and used. A control chart is able to display trends in the performance of the system and when these trends are detected they can often be addressed before more serious events occur.

### 13.1 General Policy for Control Charts

- 1. Control chart must reflect the actual performance of the system. What is charted must be relevant to the system performance. The information must be timely so that trends can be detected in a proactive manner before the process gets completely out of control and a large amount of unacceptable "product", is produced. For an environmental testing laboratory the product is analytical results. These results must be accurate, precise, representative, and comparable to be useful to the client.
- 2. Presently the NET Cambridge control chart program is based on laboratory control samples (LCS) and method blank surrogate recoveries. An LCS is a clean sample matrix spiked with (or containing) a known amount of certain target analytes. The advantage of using an LCS is that the LCS is carried through the entire analytical process, both samples preparation and analysis, along with the client samples. The recovery of target analytes in the LCS is indicative of the performance of the analytical system, apart from any effects from field sample matrices. For metals, mercury, wet chemistry, pesticides, and PCBs the LCS containing target analytes or elements is used. For volatiles and semivolatiles the surrogates in the blank are plotted on the control charts.
- 3. Another important aspect of the NET Cambridge control chart program is analyst involvement. To be truly useful, the personnel involved with carrying out the analyses must know how the system is performing. By this direct involvement they will participate in detecting out-of-control conditions and will be actively responsible for correcting any conditions causing the analytical system to perform poorly.

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### 13.2 Types of Control Charts Used

A control chart is a graph of a quality control parameter over time. As currently defined the Control Chart Program will use what are referred to as "X" charts since the individual observations are plotted. The charts will contain all LCS or blank surrogate recoveries for a given analysis and matrix (water or soil) with no averaging or sub-sampling. All out of control LCS values are to be included on the control charts but will not be used in the calculation of limits if there is an assignable cause. In addition to the individual data points, and a line connecting these points, the chart will also contain the following horizontal lines:

- Upper control limit (UCL) -- the mean plus three standard deviations as calculated from a representative set of 25 data points
- Lower control limit (LCL) -- the mean minus three standard deviations as calculated from a representative set of 25 data points

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### 13.3 Analyses Charted

- Metals EVERY LCS for all furnace metals, at least 3 ICP metals, and mercury (except those LCSs that are associated with unacceptable calibration QC) each matrix plotted separately
- plotted by digestion date and digestion batch ID
- CN EVERY CYANIDE LCS recovery for each matrix separately (Since there are many ways to do cyanide we have decided to plot only the types of analyses that are used for CLP type work, i.e., no physiological or DEP cyanide).
- Plotted by preparation/analysis date
- VOA ALL 3 SURROGATES IN EVERY METHOD BLANK ON EVERY INSTRUMENT, each matrix plotted separately,
- plotted by run file ID indicating instrument and date of analysis
- SemiVoa ALL 6 SURROGATES IN EVERY METHOD BLANK ON EVERY INSTRUMENT, each matrix plotted separately
- plotted by extraction date
- Pest. EVERY LCS RECOVERY OF DDT AND HEPTACHLOR except those associated with unuseable initial calibrations, each matrix plotted separately
- plotted by extraction date
- PCBs EVERY LCS RECOVERY OF PCB 1260 and 1016 except those associated with unuseable initial calibrations, each matrix plotted separately
- plotted by extraction date
- TPH EVERY LCS RECOVERY except those associated with unuseable initial calibrations, each matrix plotted separately
- plotted by extraction date

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### 13.4 Generating Control Charts

Control charts are prepared by using SQC a statistical software accessed through the network There are several choices in the Software which affect the style and content of control charts. The user is allowed some flexibility but all control charts must meet the following criteria:

- The vertical axis (y-axis) of the chart is for the LCS recovery, in percent. The range must encompass the lower and upper allowable limits as defined by the analytical method. The horizontal axis (x-axis) is for the date of LCS preparation (or analysis for volatiles).
- Each chart must be identified by the analyte or element being plotted and the appropriate matrix.
- There should be only one LCS type per chart.
- The points on the chart must be identified by date and be accompanied by their corresponding recovery value.

### 13.4 Interpreting Control Charts

As the chart is updated, the person preparing the chart is responsible for examining the chart for the out-of-control conditions defined below. Any out-of-control conditions are to be reported immediately to the Supervisor, and a documented corrective action investigation must follow.

- Any one point is outside the control limits (above the UCL or below the LCL).
- Any seven consecutive points are on the same side of the mean.
- Any seven consecutive points are successively larger, or smaller, than its immediate predecessor.
- Any obvious cyclic pattern is seen in the points.

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### 13.5 Review of Control Charts

On a weekly basis, the chart is to be reviewed by the Supervisor. This will include seeing that the chart is up to date, is being properly prepared, and that no out-of-control conditions have been overlooked by the analyst.

On a monthly basis, the chart is to be reviewed by the Section Manager. This will include seeing that the chart is up to date, is being properly prepared, and that no out-of-control conditions have been overlooked by the analyst. The Section Manager will ensure that a laboratory non-conformance report is filed for any out-of-control conditions. At this time the Section Manager will ensure that the current control charts are provided to the QAC for inclusion in any required internal or external QA reports (e.g. Monthly Progress Reports).

On a quarterly basis, the Control Chart Program will be reviewed by the QAC and the Laboratory Director. At this time the need for changes in the program will be considered.

### 13.6 Responding to Out of Control Conditions

All out of control conditions noted from control charts need to be documented, reported, and investigated. The proper format for this report is the "Laboratory Non-Conformance Report".

Only LCS or blank surrogate recoveries that exceed the allowable limits defined by the method protocol will result in decisions to reject sample data. These recoveries should still included in the control charts, with an annotation that this point should not be included in any recomputed control limits.

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### 14.0 CORRECTIVE ACTION

important part of any quality assurance program is well-defined well-defined effective policy for correcting quality problems. NET Cambridge employs a corrective action system which operates under the direction of the Division OA Coordinator. While the entire quality assurance program is designed to avoid problems, it also serves to identify and correct the problems that do exist. Usually these quality problems fall into two categories, immediate corrective action or long-term corrective action.

### 14.1 Immediate Corrective Action

Specific quality control limits are in place to help analysts recognize the need for corrective action. Any quality control indicators that are outside of the acceptable criteria presented in Table 7.1 require some form of corrective action. Often an analysts experience will allow him/her to initiate immediate corrective action at the bench All that is required in these instances is that the level. incident be documented in the sample preparation or analysis record.

### 14.1 Long Term Corrective Action

The need for more formal corrective action may be identifie. by performance evaluation sample results, control chart trends, or internal or external audits. Any quality problem which cannot be solved by immediate corrective action falls this category. The Division Quality Coordinator is responsible for managing the corrective action process for long term corrective actions. The Quality Control Coordinator may, with the support of the Division Manager, delegate responsibilities for investigating problems and implementing solutions to appropriate operational groups or individuals.

The essential steps in the closed loop corrective action system are:

- identification of the problem;
- assignment of responsibility for investigating the problem;
- determination of the cause of the problem through investigation;
- formulation of a corrective action plan;
- monitoring the effectiveness of the corrective action plan;
- verifying the elimination of the problem; and
- documenting the processes involved in the above.

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### 14.3 Documentation of Corrective Actions

The documentation of corrective actions and the maintenance of all corrective action reports is the responsibility of the QA Coordinator. The documentation consists of a logbook containing the following:

- corrective action requests (see Figure 14.1)
- a brief description of the problem
- analyses or operation affected
- location of further documentation such as a case or job folder
- date when corrective action completed

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### 15.0 DATA EVALUATION, AND REPORTING

### 15.1 Data Reduction

Analysis results will be reduced to the concentration units specified in the analytical procedures using the equations provided in the analytical references listed in Section 9. These calculations are an integral part of the analysis and as such are the responsibility of laboratory analysts. All calculations will be checked by laboratory supervisors. Laboratory managers will routinely check approximately 10% of all data for valid QC. The full data set is checked for completeness by the project manager.

#### 15.2 Data Evaluation

Data evaluation is the process by which analytical data are accepted or rejected based on a set of criteria. NET-Cambridge personnel use the following criteria in the evaluation of laboratory data:

- use of published or approved analytical procedures;
- use of properly operating and calibrated instruments;
- precision and accuracy achieved comparable to that achieved in similar analytical programs;
- precision, accuracy and blank contamination meeting project specific criteria outlined in Table 5.2 and 5.3;
- completeness of the data set.

All data will be evaluated by laboratory supervisors prior to being released for reporting purposes to the NET project manager. The persons evaluating the data will have sufficient knowledge of the technical work to identify questionable values. All analyses requiring CLP protocols will be evaluated in accordance with the requirements of those protocols. Occasionally a result is found that does not meet the specified criteria for evaluation. The reporting of such a result and its associated samples is decided on a case by case basis by the laboratory manager, who is also responsible for providing a narrative documenting the event and the decision.

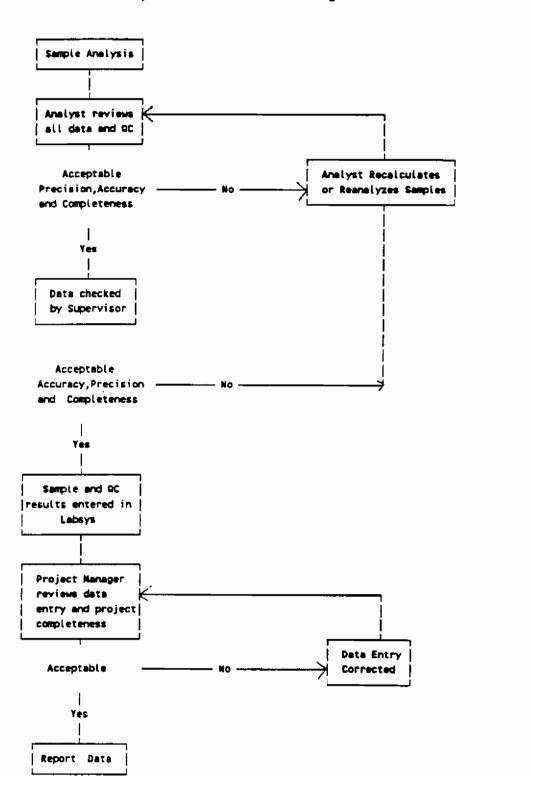
### Data Review Process 15.3

The flow charts below depicts the analytical data reduction, evaluation and reporting process. Personnel who will handle data gathering and evaluation are shown in the Division Organization Chart (Figure 1.1).

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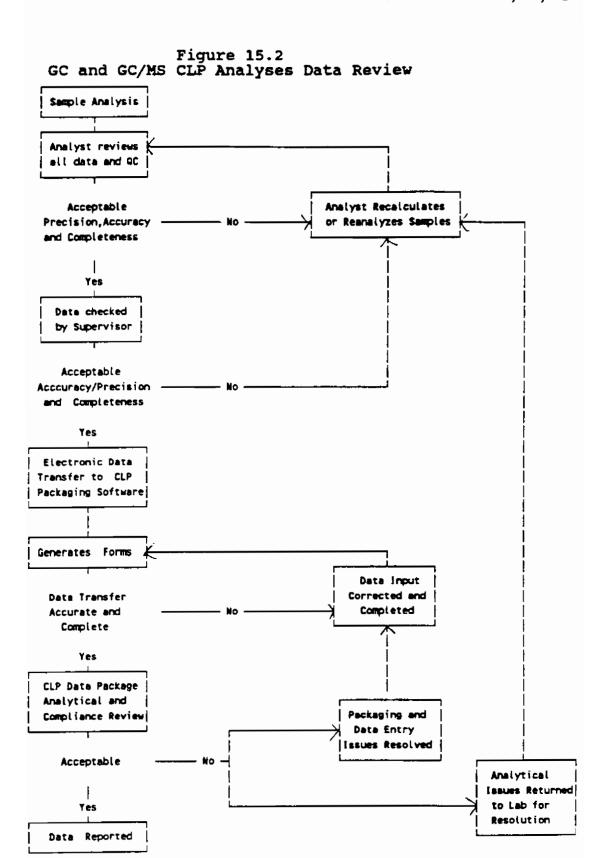
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Figure 15.1 GC and GC/MS Commercial Analysis Data Review



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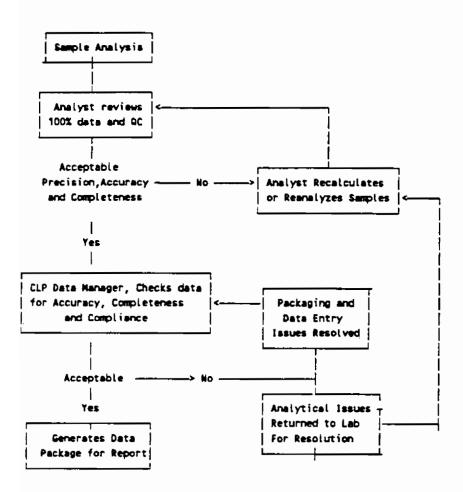
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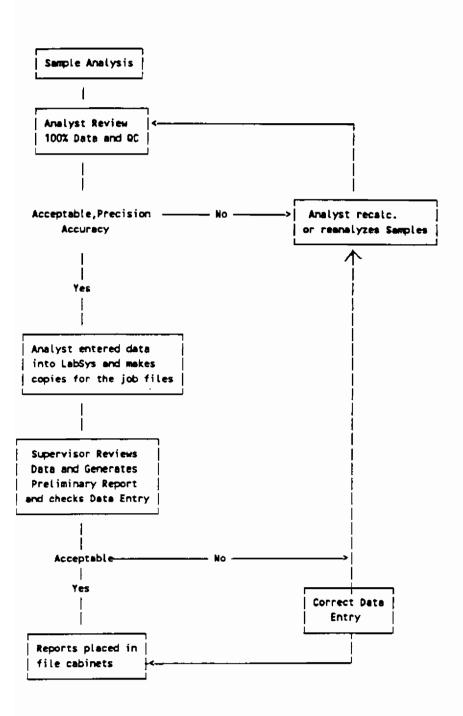
Figure 15.3 Metals CLP Data Review



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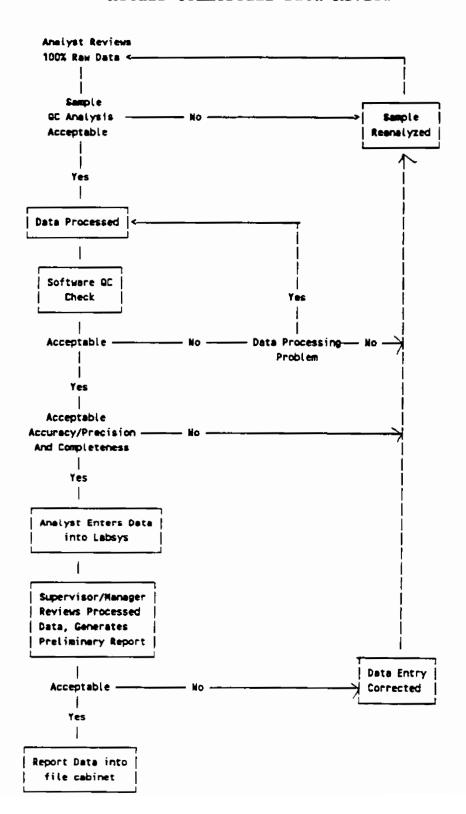
Figure 15.4 Conventional Chemistry Data Review



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Figure 15.5
Metals Commercial Data Review



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### 15.4 Data Reporting

NET uses a Laboratory Information Management System for tracking and reporting of analysis data.

A standard report includes:

- o cover page;
- o sample results;
- o statement of methods for each parameter;
- o date of sample receipt;
- o initialed chain of custody form;
- o minimum detection limits for each method;
- o sample extraction and analysis dates; and
- o case narrative summarizing any problems or corrective actions associated with the case.

CLP data are reported using CLP specified forms and in CLP protocol format. NET Cambridge uses a custom designed data management system for reporting CLP inorganics data. This system transfers data from dedicated microprocessors on each instrument to a central computer for storage and processing. CLP Organics data are screened for compliance by Finnigan's Q/A-Formaster II system which produces reports in CLP organics format.

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### 16. AUDITS AND APPROVALS

### 16.1 System Audits

A system audit is an evaluation of the laboratory's quality assurance practices and operating procedures. This audit consists of an on-site review of the laboratory's quality assurance systems and its physical facilities. In addition to NET's own internal system of periodic inspection by the Cambridge QA Coordinator, and the NET Corporate Quality Director, system audits are performed on a regular basis by thgovernment agencies and clients. NET-Cambridge will cooperate with all on-site external systems audits performed by the client.

The audit may include several or all of the components listed below:

- o Personnel, facilities and equipment;
- o Chain-of-custody procedures;
- o Instrument calibration and maintenance;
- o Standards preparation and verification;
- o Analytical procedures;
- o Quality control procedures;
- o Data handling procedures;
- Documentation control procedures.

### 16.2 Performance Audits

Performance audits provide a systematic check of laboratory data quality and measurement systems. For maximum usefulness two types of performance evaluation samples are employed, single blind and double blind.

<u>Single-blind</u> - a sample which is known by all concerned to be a PE and only the values are unknown. The results of these samples are useful in determining technical systemic problems within the operating group.

<u>Double-blind</u> - a sample that appears to be a client sample its identity and values are both unknown to the laboratory. Double blind samples are useful in identifying technical systemic problems, random analytical problems, and non-technical systemic problems.

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NET-Cambridge routinely participates in single-blind laboratory performance evaluations for the U.S. EPA as part of the Water Supply (WS) and Water Pollution (WP) programs, and similar programs administered by the State of New York. NET-Cambridge participates in an Interlaboratory Testing Program (ITP) as part of its corporate quality assurance program. Additional performance evaluations are submitted on client, contract and project specific bases.

A schedule for NET's participation in these single-blind and double-blind performance audits is detailed in Table 16.1. The reports from these audits will be made available if requested by the client.

Table 16.1

Annual Laboratory Performance Evaluation Schedule

PE Set	1st quarter	2nd quarter	3rd quarter	4th			
EPA WS		x	,				
EPA WP	x		x				
NYDOH	Non-Potables	Potables	Non-Potables				
CLP *							
Organic	x	x	x	x			
Inorganic	x	x	x	x			
Organic ITP	x		x				
Inorganic II	P X		x				

NYSDOH - New York State Department of Health

* CLP quarterly blind samples analyzed when made available through the EPA regional offices.

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### 16.3 Approvals

NET Cambridge is certified to perform analyses on wastewater, drinking water and soil matrices in several states and for several government programs. Table 16.2 lists the certifications or approvals currently held by NET Cambridge. An NET Project manager will provide a list of certified parameters for states of interest upon request.

In addition to state certifications NET Cambridge has been approved to conduct analyses for the following government programs:

- US Army Corps of Engineers Defense Environmental Restoration Program
- Hazardous Waste Remedial Action Program HAZWRAP
- Navy Energy & Environmental Support Activity NEESA

Table 16.2 State Certifications

State	Potable Water	Non-Potable
Connecticut	Y	Y
Florida	N	Y
Maine	Y	N
Massachusetts	Y	Y
New Hampshire	Y	Y
New Jersey	Y	Y
New York	Y	Y
Pennsylvania	Y	N
Rhode Island	Y	Y
South Carolina	Y	Y
Virginia	Y Y	N

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### 17. QUALITY ASSURANCE REPORT TO MANAGEMENT

In order to provide data and service of consistently high quality there must be frequent and timely communication of quality con-cerns to the operational groups and senior management. The Quality Assurance Coordinator also prepares a monthly report of quality issues for the Division Manager and the Corporate Director of Data Quality. These reports include:

- results of performance evaluation studies for ongoing or 0 new contracts;
- Control charts of internal QC sample results analyzed each 0 month
- Internal and external system audit reports and corrective 0 action responses;
- updated information on state or program certifications 0
- status on the development and implementation of National 0 and Division specific SOPs;
- significant quality assurance problems and recommended 0 solutions.

# ENVIRONMENTAL COMPLIANCE BRANCH STANDARD OPERATING PROCEDURES AND QUALITY ASSURANCE MANUAL FEBRUARY 1, 1991

U.S. Environmental Protection Agency Region IV Environmental Services Division College Station Road Athens, Georgia 30613

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### 3.0 SAMPLE CONTROL, FIELD RECORDS, AND DOCUMENT CONTROL

### 3.1 INTRODUCTION

The objectives of this section are to present the Branch standard operating procedures for sample identification, sample control and chain-of-custody, maintenance of field records, and document control.

A sample is defined as physical evidence collected from a facility, site, or the environment. For the purposes of this section, the term "physical evidence" also includes photographs, records, or any other tangible article collected from the environment, facility, or site.

All sample identification, field records, and chain-of-custody records shall be recorded in waterproof, non-erasable ink. If errors are made in any of these documents, Branch personnel will make corrections by simply crossing a single line through the error and entering the correct information. All corrections shall be initialed and dated by the investigator. If possible, all corrections should be made by the individual making the error.

If information is entered onto sample tags, logbooks, or sample containers utilizing stick-on labels, these labels should not be capable of removal later without leaving obvious indications of the attempt. Labels should never be placed over previously recorded information. Corrections to information recorded on stick-on labels should be made as stated in the previous paragraph.

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### 3.2 SAMPLE AND EVIDENCE IDENTIFICATION

### 3.2.1 General

The method of sample identification utilized depends on the type of sample collected. Samples collected for in-situ field analyses are those collected for specific field analyses or measurements where the data are recorded directly in bound field logbooks or recorded directly on the Chain-of-Custody Record, with identifying information, while in the custody of the sampling team. Examples of such in-situ field measurements and analyses include pH, temperature, and conductivity. Also included in this category are those field measurements or analyses such as flow measurements, geophysical measurements, surveying measurements, etc. that are made with field instruments or analyzers, where no sample is actually collected.

### 3.2.2 Sample Identification

Samples, other than those collected for in-situ field measurements or analyses, are identified by using a standard sample tag (Figure 3.2.1) which is attached to the sample container. In some cases, particularly with biological samples, the sample tag may have to be included with or wrapped around the sample. The sample tags are sequentially numbered and are accountable documents after they are completed and attached to a sample or other physical evidence. The following information shall be included on the sample tag:

- ESD project number;
- field identification or sample station number;
- date and time of sample collection:
- designation of the sample as a grab or composite;
- type of sample (water, wastewater, leachate, soil, sediment, etc.)
   and a very brief description of the sampling location;
- the signature(s) of the sampler(s) or of the designated sampling team leader (a team leader is a field investigator assigned by the project leader to be present during the collection of a specific sample and to be responsible and knowledgeable of all activities directly related to the collection of that sample).
- whether the sample is preserved or unpreserved;
- the general types of analyses to be conducted (checked on front of tag); and
- any relevant comments (such as readily detectable or identifiable odor, color, or known toxic properties).

Samples or other physical evidence collected by Branch personnel during criminal investigations are to be identified by using the "criminal sample tag."

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This tag is identical to the standard sample tag shown in Figure 3.2.1, except that it has a red border around the front and a red background on the back of the tag.

The field sample station number is assigned by the project leader or field investigator. This number is ordinarily an alpha-numeric code, designed for a particular inspection or investigation. For example, if a sample is collected from a monitoring well installed during a site screening investigation conducted at the Abercrombie Widget Company, the alpha-numeric sample number code could be AW-001W. A surface soil sample from this facility might be identified as AW-002S. Each separate monitoring location should have a different numerical designation. Frequently, water and sediment samples are collected from the same sampling station and could have the same numerical designation. For example, water and sediment samples collected from the same location in the Oconee River at Station 001 would be identified as OR-001W and OR-001S, respectively. The project leader or field investigator shall exercise due caution to insure that sample station numbers are not duplicated during studies. The exact description of all sampling stations associated with field identification or sample station numbers shall be documented in the bound field logbooks.

If a sample is split with a facility, state regulatory agency, or other party representative, sample tags or labels with identical information should be attached to each of the sample containers by the party receiving the split sample. Also, all tags for blank or duplicate samples will be marked "blank" or "duplicate," respectively. This requirement does not apply to "blind" spiked or blank samples which are to be submitted for laboratory quality control purposes. "Blind" spiked or blank samples shall not be identified as such, but will be submitted as outlined in Section 4.6.9. This identifying information shall also be recorded in the bound field logbooks and on the Chain-Of-Custody Record as outlined in Section 4.4.

### 3.2.3 Photograph Identification

All photographs taken by Branch personnel shall be identified on the back of the print with the following information:

- an accurate description of what the photograph shows, including the name of the facility or site and the location.
- date, time and the location that the photograph was taken;
- the orientation of the photograph (i.e., looking northeast, etc.);
- the signature of the photographer.

If the photograph was taken with a Polaroid camera, the information shall be entered on the back of each photograph as soon as it is taken. If a 35 mm camera is used, a serial type record of each frame exposed shall be kept in the bound field logbook along with the information required for each photograph. The film shall be developed with the negatives supplied uncut. The field investigator shall then enter the required information on the prints, using the serialized photographic record from the bound field logbook, to identify each

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photograph. For criminal investigations, the negatives must be maintained with the bound field logbook in the project file and stored in the secured file cabinet.

### 3.2.4 Identification of Physical Evidence

Physical evidence, other than samples, shall be identified by utilizing a sample tag or recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (barrels for example), the field investigator shall mark the vessel or container with the field identification or sample station number for future identification, if necessary. The vessel or container shall be marked by utilizing a sharp instrument or spray paint. The vessel or container need not be marked if it already has a unique marking or serial number; however, these numbers shall be recorded on the sample tag and in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. Field investigators should mark the charts and write the following information on these charts while they are still in the instrument or recorder (along with the field investigators initials):

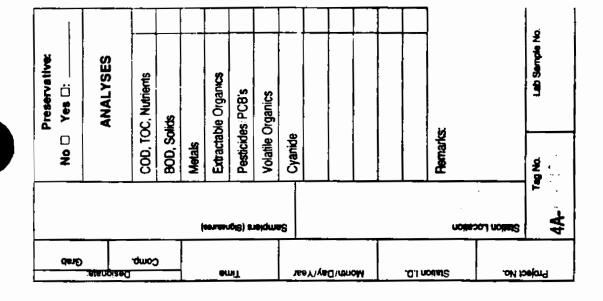
- if possible, starting and ending time(s) and date(s) for the chart;
- if possible, the field investigator shall take an instantaneous measurement of the media being measured by the recorder. The instantaneous measurement shall be entered at the appropriate location on the chart. The field investigator shall enter the date and time of the measurement and then enter his/her initials:
- a description of the location being monitored and any other information required to interpret the data such as type of flow device, chart units, factors, etc.

After the chart has been removed, the field investigator shall indicate on the chart who the chart (or copy of the chart) was received from and enter the date and time, as well as the investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages, and from whom they were received. Branch personnel shall not accept confidential documents, except in special circumstances such as process audits, hazardous waste site investigations, etc.

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FIGURE 3.2.1 SAMPLE TAG



### FRONT

### UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 4
Environmental Services Division
College Station Road
Athens, GA 30613-7799





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### 3.3 CHAIN-OF-CUSTODY PROCEDURES

### 3.3.1 General

The possession of samples or other physical evidence shall be traceable from the time they are obtained until they are introduced as evidence in legal proceedings. This section covers only that portion of the Environmental Services Division chain-of-custody procedures that are the responsibility of Branch personnel.

### 3.3.2 Sample Custody

A sample or other physical evidence is in custody if:

- it is in the field investigator's or the transferee's actual possession; or
- it is in the field investigator's or the transferee's view, after being in his/her physical possession; or
- it was in the field investigator's or the transferee's physical possession and then he/she secured it to prevent tampering; or
- it is placed in a designated secure area.

### 3.3.3 Chain-of-Custody Record

The field Chain-Of-Custody Record (Figure 3.3.1) is used to record the custody of all samples or other physical evidence collected and maintained by Branch personnel. This form <u>shall not</u> be used to document the collection of split or duplicate samples where there is a legal requirement to provide a receipt for samples (see Section 3.4). The Chain-Of-Custody Record also serves as a sample logging mechanism for the Analytical Support Branch (ASB) sample custodian.

The following information must be supplied in the indicated spaces (Figure 3.3.1) in detail to complete the field Chain-Of-Custody Record.

- The ESD project number.
- The project name.
- All samplers and /or sampling team leader must sign in the designated signature block.
- The sampling station number, date, and time of sample collection, grab or composite sample designation, and a brief description of the type of sample and the sampling location must be included on each line (each line shall contain only those samples collected at a specific location).

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- The sampling team leader's name should be recorded in the right or left margin of the Chain-Of-Custody Record when samples collected by more than one sampling team are included on the same form. The sampling team leader is an individual designated by the project leader to be responsible for all activities related to the collection of samples by a specific team of sampling personnel.
- The total number of sample containers must be listed in the indicated space for each sample. The total number of individual containers must also be listed for each type of analysis under the indicated media or miscellaneous columns. Note that it is impossible to have more than one media type per sample. The type of container and required analyses should be circled as indicated on the Record.
- The tag numbers for each sample and any needed remarks are to be supplied in the indicated column.
- The field investigator and subsequent transferee(s) must document the transfer of the samples listed on the Record in the spaces provided at the bottom of the Record. One of the samplers documented under the sampler(s) section must be the person that originally relinquished the samples or evidence or a designated field sample custodian who receives secured samples from sampling teams and maintains these samples under secure conditions. Both the person relinquishing the samples and the person receiving them must sign the form; the date and time that this occurred must be documented in the proper space on the Record. Usually, the last person receiving the samples or evidence should be a laboratory sample custodian or other evidence clerk.
- The remarks column at the bottom of the Record is used to record airbill numbers or registered or certified mail serial numbers.

The Chain-Of-Custody Record is a serialized document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

### 3.3.4 Field Custody Procedures

- To simplify the Chain-Of-Custody Record and eliminate potential litigation problems, as few people as possible should handle the sample or physical evidence during the investigation or inspection.
- The field investigator is responsible for the proper handling and custody of the samples collected (Section 3.3.2) until they are properly and formally transferred to another person or facility.

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- Sample tags (Figure 3.2.1) shall be completed for each sample, using waterproof, non-erasable ink as specified in Section 3.3.2.
- All samples shall be sealed immediately upon collection utilizing the EPA custody seal (EPA Form 7500-(R7-75)) shown in Figure 3.3.2. The field investigator may write the date and his/her signature on the seal. This requirement shall be waived if the field investigator keeps the samples in his/her continuous custody from the time of collection until they are delivered to the laboratory analyzing the samples.
- All samples must be documented in bound field logbooks.
- A Chain-Of-Custody Record will be completed for all samples or physical evidence collected as specified in Section 3.3.3. A separate Chain-Of-Custody Record will be utilized for each final destination or laboratory utilized during the inspection or investigation.
- If chain-of-custody is required for documents received during investigations, they should be placed in large envelopes, and the contents should be noted on the envelope. The envelope shall be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for the envelope. Any time the EPA seal is broken, that fact shall be noted on the Chain-Of-Custody Record and a new seal affixed. The information on the seal shall include the field investigator's signature, as well as the date and time of sealing.
- Other physical evidence such as video tapes or other small items shall be placed in Zip-Loc® type bags or envelopes and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for these items. Any time the seal is broken, a new seal shall be affixed. The information on the seal shall include the field investigator's signature, as well as the date and time of sealing.
- In general, Branch personnel shall not accept samples from other sources unless the sample collection procedures used are known to be acceptable, can be documented, and the sample chain-of-custody can be established. If such samples are accepted by Branch personnel, a standard sample tag containing all relevant information and the Chain-Of-Custody Record, shall be completed for each set of samples.
- EPA custody seals can be used to maintain custody on other items
  when necessary by using similar procedures as those outlined previously in this section.

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### 3.3.5 Transfer of Custody and Shipment

- All physical evidence or sample sets shall be accompanied by a Chain-Of-Custody Record. When transferring the possession of samples, the individual receiving the samples shall sign, date, and note the time that he/she received the samples on the Chain-Of-Custody Record. This Chain-Of-Custody Record documents transfer of custody of samples from the field investigator to another person, to the ASB, other laboratories, or other organizational elements.
- Samples shall be properly packaged for shipment (Appendix C) and delivered or shipped to the ASB or other designated laboratory for analyses. Shipping containers shall be secured by using nylon strapping tape and EPA custody seals. The custody seals shall be placed on the container so that it cannot be opened without breaking the seals. The seal shall be signed and dated by the field investigator.
- When samples are split with a facility, state regulatory agency, or other government agency, the facility, state regulatory agency, or other government agency representative should sign the Chain-Of-Custody Record. The only exception is that a Receipt For Samples Form will be used for RCRA, TSCA, and CERCLA samples as required by the appropriate regulations (Section 3.4).
- All samples shall be accompanied by the Chain-Of-Custody Record. The original and one copy of the Record will be placed in a plastic bag inside the secured shipping container if samples are shipped. One copy of the Record will be retained by the field investigator or project leader. The original Record will be transmitted to the field investigator or project leader after samples are accepted by the laboratory. This copy will become a part of the project file.
- If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading (GBL) or Air Bill should be used. Receipts from post offices, copies of GBL's, and Air Bills shall be retained as part of the documentation of the chain-of-custody. The Air Bill number, GBL number, or registered mail serial number shall be recorded in the remarks section of the Chain-Of-Custody Record or in another designated area if using a form other than that shown in Figure 3.3.1.

### FIGURE ....1

# SEPA REGION 4 U.S. ENMRONMENTAL PROTECTION AGENCY

### CHAIN OF CUSTODY RECORD

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ENVIRONMENTAL SERVICES DIVISION COLLEGE STATION ROAD

ATHENS, GEORGIA 30613-7799

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PROJECT NAME/LOCATION						·															
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FIGURE 3.3.2 CUSTODY SEAL

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### 3.4 RECEIPT FOR SAMPLES

### 3.4.1 General

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions.

### 3.4.2 Receipt for Samples Form

The Receipt For Samples Form (Figure 3.4.1) is to be used by Branch personnel to satisfy the receipt for samples provisions of RCRA, CERCIA, and TSCA. The form also documents that split samples were offered and accepted or rejected by the owner/operator of the facility or site being investigated. The following information must be supplied and entered on the Receipt For Samples Form.

- The ESD project number, project name, name of facility or site, and location of the facility or site must be entered at the top of the form in the indicated locations.
- The sampler(s) must sign the form in the indicated location.
- The facility/site owner/operator's acceptance or rejection of split samples must be checked in the appropriate place in the Split Samples Offered section of the form. The owner/operator should be requested to initial his acceptance or rejection by the check mark and to sign his name in this block indicating that he has been offered this choice if the offer is refused.
- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), the tag numbers of samples collected which will be removed from the site, a brief description of each sampling location, and the total number of sample containers for each sample must be given. If EPA sample tags are used for split samples, these tag numbers should be recorded under the remarks column.
- The bottom portion of the form is used to document the receipt of split samples by the owner/operator of the facility or site. One of the samplers must be requested to sign and complete the information in the "transferred by" section (date and time must be entered). The owner/ operator of the site must sign the "received by" section of the form (the owner/operator must give his title, and telephone number and give the date and time he/she signed the form). If the owner/operator refuses to sign the form, the sampler(s) should note

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this fact in the owner/operator's signature block and initial this entry.

The copy of the form is to be given to the facility or site owner/operator. The Receipt for Samples Form is serialized and becomes an accountable document after it is completed. The original copy of this form must be maintained in project files.

### FIGURE 3...1

## RECEIPT FOR SAMPLES

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ENVIRONMENTAL SERVICES DIVISION
COLLEGE STATION ROAD

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### 3.5 FIELD RECORDS

Branch personnel shall use only bound field logbooks for the maintenance of field records. The standard field logbooks utilized by Branch personnel are those obtained from the General Services Administration federal supply schedule numbers 7530-00-274-5494 and 7530-00-222-3525. Other bound logbooks such as bound surveyors logbooks are acceptable so long as pages cannot be removed without tearing them out.

Preferably, a logbook should be dedicated to an individual project. The investigator's name, project name, and project code should be entered on the inside of the front cover of the logbook. All entries should be dated and time of entry recorded. At the end of each day's activity, or entry of a particular event if appropriate, the investigator should draw a diagonal line at the conclusion of the entry and initial indicating the conclusion of the entry or the days activity.

All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. All sample collection equipment (where appropriate), field analytical equipment, and equipment utilized to make physical measurements shall be identified in the field logbooks as outlined in Sections 4, 5, 6, and 7 of this SOPQAM. All calculations, results, and calibration data for field sampling, field analytical, and field physical measurement equipment shall also be recorded in the field logbooks. All field analyses and measurements must be traceable to the specific piece of field equipment utilized and to the field investigator collecting the sample, making the measurement, or analyses.

All entries in field logbooks shall be dated, shall be legible, and shall contain accurate and inclusive documentation of an individual's project activities. Since field records are the basis for later written reports, language should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. Once completed, these field logbooks become accountable documents and must be maintained as part of project files.

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#### 1.6 DISPOSAL OF SAMPLES OR OTHER PHYSICAL EVIDENCE

Disposal of samples or other physical evidence obtained during Branch investigations is conducted on a case-by-case basis. Before any samples analyzed by the ASB are disposed, ASB personnel shall contact the Branch field investigator or Branch or Section Chief, in writing, requesting permission to dispose of the samples. The samples will not be disposed until the Branch field investigator or Branch or Section Chief completes the appropriate portions of the ASB memo, signs and returns the memo to the ASB, specifically giving them permission to dispose of the samples. Branch personnel should check with the EPA Program Office requesting the inspection or investigation before granting permission to dispose of samples or other physical evidence. The following general guidance is offered for the disposal of samples or other physical evidence:

- No samples, physical evidence, or any other document associated with a criminal investigation shall be disposed without written permission from EPA's Office of Criminal Investigation, the Office of Regional Counsel, or the Department of Justice.
- Quality assurance samples are routinely disposed after the analytical results are reported. ASB does not advise the Branch on the disposal of these samples.
- Samples associated with routine Branch inspections may be routinely disposed.

After samples are disposed, the ASB shall send the sample tags to the Branch field investigator. These sample tags are accountable and must be placed and maintained in the project files.

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#### 3.7 DOCUMENT CONTROL

The term document control, as it applies to Branch inspections and investigations, refers to the maintenance of inspection and investigation project files. All project files shall be maintained by the appropriate Section Secretary. All documents as outlined below shall be kept in project files. Branch personnel may keep their own files, however, all official and original documents relating to Branch inspections and investigations shall be placed in the official project files. The following documents shall be placed in the project file:

- a copy of the study plan;
- original Chain-Of-Custody Records and bound field logbooks;
- a copy of the Receipt For Sample Forms;
- all records obtained during the investigation;
- a complete copy of the analytical data and memorandums transmitting analytical data;
- sample tags from samples that have been disposed of by the ASB;
- all official correspondence received by or issued by the Branch relating to the investigation including records of telephone calls;
- one copy of the draft report (without review comments; however, peer review clearance forms shall be included);
- one copy of the final report and transmittal memorandum(s); and
- any other relevant documents related to the original investigation/ inspection or follow-up activities related to the investigation/ inspection.

Under no circumstances are any personal observations or irrelevant information to be filed in the official project files. The project leader or field investigator shall review the file at the conclusion of the project to insure that it is complete.

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#### 4.0 SAMPLING PROCEDURES

#### 4.1 INTRODUCTION

This section discusses the standard practices and procedures utilized by Branch personnel during field operations to ensure the collection of representative samples. All sampling activities conducted by Branch personnel are conducted with the expectation that they will be used for enforcement purposes, unless specifically stated to the contrary in advance of the field investigation. Therefore, the use of proper sampling procedures cannot be over emphasized. The collection of representative samples depends upon:

- ensuring that the sample taken is representative of the material or medium being sampled;
- using proper sampling, sample handling, preservation, and quality control techniques;
- properly identifying the collected samples and documenting their collection in permanent field records (field log books, Chain-Of-Custody Records); and
- maintaining sample chain-of-custody.

The objectives of this section are to present:

- general considerations that must be incorporated in all sampling operations conducted by the Branch;
- Branch sampling site selection and collection procedures for an individual medium:
- Branch sampling quality assurance procedures; and
- equipment calibration and maintenance requirements for Branch sampling equipment.

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#### 4.2 GENERAL CONSIDERATIONS

The following factors and procedures shall be considered and implemented in planning and conducting all Branch sampling operations. All these factors and procedures must be considered in view of specific objectives and scope of each individual field investigation.

## 4.2.1 Selection of Representative Sampling Sites

Representative sampling sites are dependent on the type of investigation undertaken and are discussed under type of sample procedures for each medium later in this section.

## 4.2.2 Selection and Proper Preparation of Sampling Equipment

The type of sampling equipment to be used is dictated by the investigation and is discussed for each medium later in this section. Appendix B describes the standard equipment cleaning procedures.

## 4.2.3 Sampling Equipment Construction Material

The material that sampling equipment is constructed of can affect sample analytical results. Materials used must not contaminate the sample being collected and must be readily cleaned so that samples are not cross-contaminated. The standard materials for sampling equipment used to collect samples for trace organic compounds or metals analyses are, in order of decreasing desirability; Teflon®, glass, stainless steel, and steel.

## 4.2.4 Selection of Parameters to be Measured

Parameters to be measured are usually dictated by the purpose of an investigation and should be based on required monitoring conditions (NPDES or RCRA permits for example) or on the field investigator's or requestor's knowledge of the problem being investigated.

#### 4.2.5 Required Sample Volumes

The volume of sample obtained should be sufficient to perform all required analyses with an additional amount collected to provide for quality control needs, split samples, or repeat examinations. When using a peristaltic pump, individual aliquots of a composite sample should be at least 100 milliliters in order to minimize sample solids bias.

Although the volume of sample required by contract laboratories depends on the analyses to be performed, the amount of sample required for a complete water or wastewater analysis is normally two gallons (7.6 liters) for each laboratory receiving a sample. The amount of soil/sediment required for a complete analysis is approximately 16 ounces. However, the laboratory receiving the sample should be consulted for any specific volume requirements.

The volumes of samples collected from waste sources at hazardous waste

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sites or samples from sources which are known to be toxic should be kept to an absolute minimum.

Sample volumes to be collected by Branch personnel for routine analyses by the Environmental Services Division (ESD), Analytical Support Branch (ASB) laboratory are in Appendix A. The sample volume required for each analysis is the volume of the standard container less ullage (empty space) required for sample mixing by laboratory personnel and safe shipment of samples to the laboratory. Branch personnel shall allow a minimum of ten percent ullage in every sample container for this purpose. The only exception is samples collected for purgeable organic analyses (VOA) or dissolved gases such as sulfides for which sample containers must be completely filled.

## 4.2.6 Selection and Proper Preparation of Sample Containers

The type of sample container is dictated by the analyses required. Standard sample containers used by Branch personnel are presented in Appendix A. Special sample container preparation requirements are in Appendix B.

#### 4.2.7 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected by Branch personnel are given in Appendix A. All chemical preservatives used by Branch personnel will be supplied by the ASB. All samples requiring preservation should be preserved immediately upon collection in the field. Samples that should not be preserved in the field are:

- Samples collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials. Barrel, drum, closed container, spillage, or other source samples from hazardous waste sites are not to be preserved with any chemical. These samples may be preserved by placing the sample container on ice, if necessary.
- Samples that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved using the procedures given in Appendix A.
- Samples for metals analyses which are shipped by air shall not be preserved with nitric acid in excess of the amount specified in Appendix C.
- Samples for purgeable organic compounds analyses which are shipped by air shall not be preserved with hydrochloric acid in excess of the amount specified in Appendix C.

All samples preserved with chemicals shall be clearly identified by indicating on the sample tag that the sample is preserved. If samples normally requiring preservation were not preserved, field records shall indicate why.

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### 4.2.8 Sample Holding Times

The elapsed time between sample collection and initiation of laboratory analyses must be within a prescribed time frame for each individual analysis to be performed. Sample holding times for all routine samples collected by Branch personnel are shown in Appendix A.

## 4.2.9 Special Precautions for Trace Contaminant Sampling

Some contaminants can be detected in the parts per billion and/or parts per trillion range. Extreme care must be taken to prevent cross-contamination of these samples. The following precautions shall be taken when trace contaminants are of concern:

- A clean pair of new, disposable gloves will be worn each time a different location is sampled and gloves should be donned immediately prior to sampling.
- Sample containers for source samples or samples suspected of containing high concentrations of contaminants shall be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.
- If possible, ambient samples and source samples should be collected by different field teams. If different field teams cannot be used, all ambient samples shall be collected first and placed in separate ice thests or shipping containers. Samples of waste or highly contaminated samples shall never be placed in the same ice chest as environmental samples. It is good practice to enclose waste or highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers for source samples or samples suspected to contain high concentrations of contaminants shall be lined with new, clean, plastic bags.
- If possible, one member of the field team should take all the notes, fill out tags, etc., while the other members collect all of the samples.
- When sampling surface waters, the water sample should always be collected before the sediment sample is collected.
- Sample collection activities should proceed progressively from the suspected least contaminated area to the suspected most contaminated area.
- Branch personnel should use equipment constructed of Teflon®, stainless steel, or glass that has been properly precleaned (Appendix B) for collecting samples for trace metals or organic compounds analyses. Teflon® or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC shall not be used to collect samples for trace

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organic compounds analyses.

## 4.2.10 Sample Handling and Mixing

After collection, all sample handling should be minimized. Branch personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that melted ice cannot cause sample containers to become submerged, as this may result in sample cross-contamination. Plastic bags, such as Zip-Lock® bags, should be used when small sample containers (e.g., VOA's or bacterial samples) are placed in ice chests to prevent cross-contamination.

Once a sample has been collected, it may have to be split into separate containers for different analyses. The best way to split liquid samples is to continually stir the sample contents with a clean pipette or precleaned Teflon® rod and allow the contents to be alternately siphoned into respective sample containers using Teflon® or PVC (Tygon® type) tubing. Teflon® must be used when analyses for organic compounds or trace metals are to be conducted. Any device used for stirring, or tubing used for siphoning, must be cleaned in the same manner as other equipment (Appendix B). However, samples collected for purgeables organic compounds analyses may not be split using this procedure.

A true split of soil, sediment, or sludge samples is almost impossible to accomplish under field conditions. The higher the moisture content, the more difficult it is to split the sample.

It is extremely important that soil samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample interval. The most common method of mixing is referred to as quartering. The soil in the sample pan is divided into quarters. Each quarter is mixed, then all quarters are mixed into the center of the pan. This procedure is followed several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion and occasionally turning the material over. Soil and sediment samples collected for purgeable organic compounds analyses should not be mixed. The 2-ounce (60-ml) sample container should be filled completely; no head space should remain in the sample containers.

## 4.2.11 Purgeable Organic Compounds Sampling (VOA)

Water samples to be analyzed for purgeable organic compounds should be stored in 40-ml septum vials with screw cap and Teflon®-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the sampling program.

The vials (40-ml) should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface

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tension holds the water in a "convex meniscus." The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If any bubbles are present, repeat the procedure. Since the VOA vials are pre-preserved, extreme caution should be exercised when the vials are used as the collection device for surface water samples in order to prevent the loss of the preservative. When collecting water samples for purgeable organic compounds, triplicate samples should always be collected from each location. Three 40-ml vials containing four drops of concentrated HCl should be filled the with sample.

#### 4.2.12 Sample Identification

All samples will be fully documented, as outlined in Section 3, in the field records, on the field sample Chain-Of-Custody Record, and on the sample tags.

## 4.2.13 Procedures for Identifying Potentially Hazardous Samples

Any sample either known or thought to be hazardous should be so identified on both the sample tag and the field sample Chain-Of-Custody Record. Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed.

## 4.2.14 Collection of Auxiliary Data

All auxiliary data such as flow measurements, photographs of sampling sites, meteorological conditions, and other observations shall be entered onto the field records when the auxiliary data are collected. Auxiliary data relative to a particular sampling location should be collected as close to the sample collection time as possible. Specific types of auxiliary data to collect for each medium sampled are discussed later in this section.

## 4.2.15 Time Records

All records of time shall be kept using local time in the 2400 hour time format and shall be recorded to the nearest minute.

#### 4.2.16 Transporting and Shipping of Samples

Samples may be hand delivered to the laboratory using government owned (or rented) vehicles or they may be shipped by common carrier. All Branch personnel must be aware that certain samples are hazardous materials and, as such, are regulated by the U. S. Department of Transportation under the Transportation Safety Act of 1974. These regulations are contained in Title 49, CFR, Parts 110-119. Shipment of dangerous goods by air cargo is also regulated by the United Nations/International Civil Aviation Organization (UN/ICAO). The Dangerous Goods Regulations promulgated by the International Air Transport Association (IATA) meet or exceed DOT and UN/ICAO requirements and should be used for shipment of dangerous goods via air cargo. Shipment of samples by EPA employees is also governed by EPA Order No. 1000.18 (1). The standard Branch procedure for shipping environmental samples is in Appendix C. All Branch employees shall meet

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the intent of the Transportation Safety Act of 1974 when shipping samples by common carrier.

## 4.2.17 Sample Chain-of-Custody

Branch employees shall maintain sample chain-of-custody during all field investigations for all samples collected. The standard sample chain-of-custody procedures used by Branch employees are given in Section 3.3.

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## 4.3 DEFINITIONS

## 4.3.1 Grab Sample

An individual sample collected from a single location at a specific time or period of time generally not exceeding 15 minutes. Grab samples are associated with surface water, groundwater, wastewater, waste, contaminated surfaces, soil, and sediment sampling. Grab samples are typically used to characterize the media at a particular instant in time. See Appendix A for additional guidance concerning parameters requiring a grab sample and for monitoring that indicates that grab samples must be collected.

### 4.3.2 Composite Samples

A sample collected over time that typically consists of a series of discrete samples which are combined or "composited". Four types of composite samples are listed below:

- <u>Time Composite (TC)</u>: A sample comprised of a varying number of discrete samples collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.
- Flow Proportioned Composite (FPC): A sample collected proportional to the flow rate during the compositing period by either a time-varying/constant volume (TVCV) or time-constant/varying volume (TCVV) method. The TVCV method is typically used with automatic samplers that are paced by a flow meter. The TCVV method is a manual method that individually proportions a series of discretely collected samples. The FPC is typically used when sampling wastewater.
- <u>Areal Composite</u>: A sample collected from individual grab samples collected on an areal or cross-sectional basis. Areal composites shall be made up of equal volumes of grab samples. Each grab sample shall be collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from grid points.
- Vertical Composite: A sample collected from individual grab samples collected from a vertical cross section. Vertical composites shall be made up of equal volumes of grab samples. Each grab sample shall be collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes and estuaries.

## 4.3.3 Quality Control Samples

The majority of ECB field studies require the collection of additional samples for various quality control purposes. These include the isolation of site effects (control samples), define background conditions (background sample), evaluate field/laboratory methodology (spikes and blanks, trip blanks, duplicate samples), or to assess sampling equipment (sampler blanks and equipment rinse

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blanks). In addition, it may be necessary to provide split or duplicate samples to assess field sampling procedures.

Miscellaneous sampling definitions are listed below:

- <u>Sample Aliquot</u>: A portion of a sample that is representative of the entire sample.
- <u>Split Sample</u>: A sample which has been portioned into two or more containers from a single sample container or sample mixing container.
- <u>Duplicate Sample</u>: Two or more samples collected simultaneously into separate containers from the same source under identical conditions.
- <u>Control Sample</u>: A sample collected upstream or upgradient from a source or site to isolate the effects of the source or site on the particular ambient medium being sampled.
- <u>Background Sample</u>: A sample collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.
- <u>Biased Sample</u>: A sample which is known to be non-representative of the entire site being studied. An example is samples collected during Superfund Site Screening Investigations that are intentionally biased towards suspected areas of contamination.
- <u>Trip Blanks</u>: Trip blanks are prepared prior to the sampling event in the actual sample container and are kept with the investigative samples throughout the sampling event. They are then packaged for shipment with the other samples and sent for analysis. At no time after their preparation are the sample containers to be opened before they reach the laboratory. ESD utilizes volatile organic trip blanks to determine if samples were contaminated during storage and transportation back to the laboratory. If samples are to be shipped, trip blanks are to be provided per shipment but not per cooler.
- Equipment Blanks: Equipment field blanks are defined as samples which are obtained by running organic-free water over/through sample collection equipment after it has been cleaned. These samples will be used to determine if cleaning procedures were adequate. (The equipment could have been cleaned in the field or prior to the field operation.)
- Pre and Post Preservative Blanks: To determine if the preservative used during field operations were contaminated, pre and post preservative blanks are prepared. On small studies, usually only a post preservative sample will be prepared. These samples are prepared by putting analyte-free/organic-free water in the container and then preserving the sample with the appropriate preservative.

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• <u>Field Blanks</u>: Organic-free water is taken to the field in sealed containers and poured into the appropriate sample containers at predesignated locations. This is done to determine if any contaminants present in the area may have an affect on the sample integrity. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

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## 4.4 DATA QUALITY OBJECTIVES

As defined in Data Quality Objectives for Remedial Response Activities (29), "Data Quality Objectives (DQO's) are qualitative and quantitative statements which specify the quality of the data required to support Agency decisions during remedial response activities". DQO's should be considered when planning any study. DQO's provide information on the limits of the data, which in turn dictate the proper uses of the data. DQO's were originally developed to guide the analytical program of Superfund (29), but the concept also has application for other field sampling efforts. Data collected in the field include samples and site information. The methods by which samples are collected may limit the uses of the subsequent analytical data. The methods by which site information, such as physical measurements, photographs, field notes, etc., are collected, may reduce their accuracy. The manner in which sampling equipment is cleaned will also affect the DQO level of the data. The various DQO levels are numbered I through V, with I being the lowest and IV the highest quality data. Level V data are collected using special or non-standard methods. Higher quality methods may be substituted for lower level work.

## 4.4.1 <u>DOO Level I</u>

Sampling equipment and sample containers must be cleaned using soap and tap water, visibly free of contamination, and free of detectable analytes using the analytical screening methods specified for the study. Use of organic vapor survey methods to determine locations or media fractions for higher level analysis is an example of DQO Level I field work. However, this technique is not appropriate for compounds that are not volatile or produce low instrument response. Data produced from such samples may not be used for other than the stated purpose.

#### 4.4.2 DOO Level II

Field methods, decontamination procedures, and sampling equipment construction materials for DQO Level II analyses are as specified elsewhere in this document. The construction materials for sampling equipment may vary if rinse blanks analyzed using the field analytical procedures show that the substituted equipment does not contribute detectable analytes, and the materials would not reasonably be expected to contribute detectable analytes. For example, it may be acceptable to use PVC sampling equipment to collect samples that are only being analyzed for metals, or to use equipment made of chrome plated material for samples being analyzed only for organic compounds. Field cleaning procedures for sampling equipment used to collect samples that will be analyzed at this DQO level may consist of:

- Soap and potable water wash with brush (steam may also be used), followed by potable water rinse.
- Water rinse. The quality of the water is determined by the contaminants of concern and the minimum quantitation limits of the analytical methods used. For example, if an atomic absorption (AA) unit is being used to analyze water samples for lead only, and the

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minimum quantitation limit is 20 ug/l, water containing up to 10 ug/l lead (one-half the minimum quantitation limit) may be used as decontamination water.

A minimum of five percent of samples collected for DQO Level II analyses should be split for DQO Level IV analysis. These samples must be representative of all samples submitted to the field laboratory.

## 4.3.3 DOO Level III

Field methods, decontamination procedures, and sampling equipment construction materials for DQO Level III analyses are as specified elsewhere in this document. Some modifications of these specifications are allowable in certain limited instances, as specified below.

If DQO Level III analytical services are being used in support of drilling or excavation operations, the cleaning procedures for the down-hole drilling or excavation equipment only may be cleaned as specified in Appendix E.9, with the omission of steps 3, 4, and 5. All other cleaning and decontamination procedures specified in that appendix apply.

When wells are constructed using materials that are not inert with respect to the contaminants being analyzed, data collected from those wells are DQO Level III or lower for those incompatible analytes, even if DQO Level IV analytical procedures are used.

A minimum of one equipment rinse blank per week for each week sampling equipment is field cleaned is required to be analyzed. If samples are preserved, a preservative blank must be collected and analyzed in the field at the beginning and end of the study. A blank of the rinse water must be collected and analyzed prior to beginning the study and at the end of each week sampling equipment is field cleaned.

A minimum of five percent of samples collected for DQO Level III analysis should be split for DQO Level IV analysis. These samples must be <u>representative</u> of all samples submitted to the field laboratory.

## 4.4.4 <u>DOO LevelaIV</u>

Field methods and equipment decontamination procedures described in this document are considered to be level IV methods. These are the standard methods to be used on all studies requiring DQO Level IV quality data. Any deviations from these methods must be documented in the field logbook or the approved study plan. The sampler must be aware that such deviations in the field work may reduce the DQO level of the data, with a subsequent reduction in the data uses. The field notes are always collected as specified in Section 3.5. Photographs are always logged as specified in Section 3.2.3.

# 4.4.5 DOO Level V

Because DQO Level V procedures are by definition non-standard, they are not

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discussed in detail. The project leader must be aware that special analytical procedures may require specialized field procedures and equipment. These must be specified in the approved study plan prior to beginning the study.

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## 4.5 INVESTIGATION DERIVED WASTE

#### 4.5.1 General

Many field investigations conducted by the Branch generate waste materials. These waste materials are known as investigation derived waste (IDW) (30). Some of these waste materials may be hazardous wastes which must be properly disposed in accordance with EPA regulations. Most, but not all, of these hazardous wastes will be associated with investigations of hazardous waste sites.

- 4.5.1.1 Types of IDW -- Materials which may become IDW requiring proper treatment, storage and disposal are:
  - Personnel protective equipment (PPE). This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
  - Disposable equipment (DE). This includes plastic ground and equipment covers, aluminum foil, conduit pipe, coliwasa samplers, Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
  - Soil cuttings from drilling or hand auguring.
  - Drilling mud or water used for water rotary drilling.
  - Groundwater obtained through well development or well purging.
  - Cleaning fluids such as spent solvent and washwater.
- 4.5.1.2 Management of Non-Hazardous IDW -- Disposal of non-hazardous IDW from hazardous waste sites should be addressed in the study plan. Non-hazardous IDW such as PPE and DE may be double-bagged and brought back to the Branch warehouse for disposal in the warehouse trash containers. To reduce volume for transportation back to the warehouse, it may be necessary to compact the waste into a reusable container, such as a 55-gallon drum. If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE and DE IDW into the facilities dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a permitted landfill local to the site. On larger studies, waste hauling services may be obtained and a dumpster located at the study site. They may also be buried on site near the contamination source, with the burial location noted in the field logbook.

Disposal of non-hazardous IDW such as drill cuttings, purge or development water, decontamination fluids, drilling muds, etc., should be specified in the approved study plan. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place the liquid IDW into the facilities treatment system. It may be feasible to spread drill cuttings around the borehole, or if the well is temporary, to replace the cuttings back into the borehole. Cuttings, purge

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water, or development water may also be placed in a pit in or near the source area. Monitoring well purge or development water may also be poured onto the ground downgradient of the monitoring well. Purge water from private potable wells which are in use may be discharged to the ground surface.

4.5.1.3 Management of Hazardous IDW -- Disposal of hazardous or suspected hazardous IDW must be specified in the approved study plan. Hazardous IDW must be disposed as specified in USEPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed of in the source area from which they originated, if doing so does not endanger human health and the environment.

If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to permit arrangements for proper containerization, labelling, transportation, and disposal/treatment in accordance with USEPA regulations.

Hazardous investigation derived waste should be kept to a minimum to conserve Branch resources. Most of the routine studies conducted by the Branch should not produce any IDW that are hazardous, with the possible exception of purged groundwater. Many of the above PPE and DE wastes can be deposited in municipal dumpsters if care is taken to keep them segregated from hazardous waste contaminated materials. Disposable equipment can often be cleaned to render it non-hazardous, as can some PPE, such as splash suits. The volume of spent solvent waste produced during equipment decontamination can be reduced or eliminated by applying only the minimum amount of solvent necessary.

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#### 4.6 SPECIFIC SAMPLE COLLECTION QUALITY CONTROL PROCEDURES

#### 4.6.1 General

This subsection provides guidelines for establishing quality control procedures for Branch sampling activities. Specific guidelines for sample site selection, selection of sampling equipment, types of samples to be collected, standard sample collection procedures, specific maintenance and calibration procedures for sampling equipment, and other considerations are presented for each medium later in this Section. Specific requirements for all sampling activities are presented in Section 4.2. Strict adherence to all of the standard operating procedures outlined in this chapter form the basis for the Branch sampling quality assurance program.

#### 4.6.2 Experience Requirements

There is no substitute for field experience. Therefore, all professional and paraprofessional Branch employees shall have the equivalent of six months field experience before they are permitted to select sampling sites on their own initiative. This field experience shall be gained by on-the-job training using the "buddy" system. Each new Branch field employee shall accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior Branch technical staff.

#### 4.6.3 Traceability Requirements

All sample collection activities shall be traceable through field records to the person collecting the sample and to the specific piece of sampling equipment (where appropriate) used to collect that sample. All maintenance and calibration records for sampling equipment (where appropriate) shall be kept so that they are similarly traceable.

#### 4.6.4 Measurement of Relative Sampling Precision

The following duplicate sampling procedures shall be used during the collection of Branch samples as a relative measure of the precision of the sample collection process. Duplicate grab and/or composite samples shall be collected during all major investigations and studies (more than 20 samples) conducted by the Branch. No more than ten percent of all samples shall be collected in duplicate. These samples shall be collected at the same time, using the same procedures, the same aquipment, and in the same types of containers as the required samples. They shall also be preserved in the same manner and submitted for the same analyses as the required samples. The collection of duplicate composite samples shall require the installation of duplicate automatic sampler setups if automatic samplers are used for sample collection.

These data will be examined by the project leader to determine if any problems are evident with specific types of media samples or with the procedures used by specific Branch personnel. The project leader will advise the Branch Quality Assurance Officer who will in turn advise the Branch and appropriate

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Section Chiefs of any problems encountered so that corrective action can be taken.

## 4.6.5 Measurement of Sample Handling Effectiveness

The effectiveness of Branch personnel sample handling techniques will be monitored by utilizing field blank samples. For routine studies these blank samples will be prepared by ASB personnel. Branch personnel shall request that these samples be provided at least one week in advance of scheduled field investigations and inspections and never (except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up by Branch personnel earlier than the morning of departure for the scheduled inspection/investigation. On larger studies, where organic-free water is generated in the field, these blank samples may be made in the field. The following field blank samples are required:

- Water Sample VOA Trip Blank -- A water sample VOA trip blank is required for every study where water samples are collected for VOA analysis. Two sealed preserved (or unpreserved if appropriate) 40-ml VOA vials will be transported to the field. These field blanks will be handled and treated by Branch personnel in the same manner as the water samples collected for purgeable organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-Of-Custody Records as trip blanks.
- Soil Sample VOA Trip Blank -- A soil sample VOA trip blank is required for every study where soil samples are collected for purgeable organic compounds analysis. One 2-oz. soil VOA vial will be transported to the field. This field blank will be handled and treated by Branch personnel in the same manner as the soil samples collected for purgeable organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-Of-Custody Records as trip blanks.

The following blanks are prepared in the field:

- Inorganic Sample Preservative Blanks -- Metals and general inorganics sample containers filled with blank water will be transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample tags and in the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. A minimum of one preservative blank should be prepared at the beginning and end of all major Branch field investigations (20 samples or more).
- <u>Automatic Sampler Blanks</u> -- The procedure for collecting automatic sampler blanks is given in Section 4.5.4. In general, cleaning procedures outlined in Appendix B should be adequate to insure sample integrity. However, it is the standard practice of the

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Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds analyses. Automatic sampler blanks for other standard analyses shall be submitted at least once per quarter.

The ASB will advise the Branch Quality Assurance Officer when trip blanks or preservative blanks are unacceptably contaminated. The Branch Quality Assurance Officer will immediately initiate an investigation to determine the cause of the problem. The results of this investigation will be promptly reported to the Branch and appropriate Section Chief so that corrective action can be initiated.

# 4.6.6 Measurement of Sample Container, Sample Equipment, and Cleaning Procedure Integrity

Specific quality control procedures are outlined in Appendix B.

## 4.6.7 Measurement of Field Generated Organic-Free Water

On larger studies, organic-free water may be prepared in the field. When this is done, a system blank should be collected prior to use and at the end of the study. If the study lasts more than one week, at least one sample per week shall be collected. Blank samples should be analyzed for all analytes of interest.

# 4.6.8 Special Quality Control Procedures for Water Samples for Extractable. Pesticide, or Herbicide Organic Compounds Analyses

Branch personnel shall submit duplicate water samples for extractable, pesticide, and/or herbicide organic compounds analyses from at least one sampling location per project and laboratory used. This sample should be collected from a location expected to be relatively free from contamination, since this sample will be used for laboratory quality control purposes. The duplicate sample should be clearly identified as "Duplicate Sample for Matrix Spike" on the sample tag, Chain-Of-Custody Record, in the field logbook, and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure shall be followed for all Branch projects where water samples are collected for the indicated organic compounds analyses, whether the samples are submitted to the ASB or the EPA CLP.

## 4.6.9 Special Quality Control Procedures for EPA Contract Laboratories

Special quality control procedures are used when samples are submitted to the EPA CLP. A series of blank and spiked samples are provided by ASB personnel that are to be submitted along with the regular samples to each contract laboratory utilized. These samples will be provided by the ASB along with the necessary CLP Traffic Report Forms for each blank and spike sample. These samples shall be submitted to the respective contract laboratories on a blind basis. Fictitious station numbers will be used on the Traffic Report Forms; the contract laboratories are not to know which samples are spikes and blanks. These samples are identified as spikes or blanks only on the copy of the Traffic Report Forms hat are returned to the ASB.

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These spike and blank samples are to be obtained from the ASB utilizing the procedures outlined in Section 4.4.5.

The ASB TPO (See Section 1.9.3.1) may request, on a case-by-case basis, that Branch personnel collect split samples (or duplicate samples if appropriate) for analysis by both the ASB and contract laboratories. The split samples are to be submitted to the ASB using established procedures. The contract laboratory involved shall not be notified in any way that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the ASB.

# 4.6.10 Special Quality Assurance Procedures for Dioxin (2.3.7.8-TCDD or Other Isomers)

All samples collected for dioxin (2,3,7,8-TCDD or isomers) analyses are analyzed by other EPA laboratories or through contract laboratories. The ASB does not conduct in-house analyses for dioxin. The ASB maintains a package that outlines all current quality control procedures to be used by Branch personnel when collecting samples for dioxin (2,3,7,8-TCDD or other isomers) analyses. The ASB must be consulted before any such samples are collected.

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## 4.7 WASTEWATER SAMPLING

## 4.7.1 General Considerations

The variety of conditions at different sampling locations require that considerable judgment be made regarding the methodologies and procedures for collection of representative samples of wastewater. Each sampling point warrants attention commensurate with its complexity. There are, however, basic rules and precautions generally applicable to sample collection. The procedures used by the Branch are generally those outlined in the NPDES Compliance Inspection Manual (2). Additional guidance is given in the agency's handbook, Monitoring Industrial Wastewater (3). Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed. Therefore, the sample should be collected near the center of the flow channel, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided; however, allowances should be made for fluctuations in water depth due to flow variations.
  - In sampling from wide conduits, cross-sectional sampling should be considered. Rhodamine WT dye (See Section 7.8.2.3 for procedures) may be used as an aid in determining the most representative sampling point(s); and
  - If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquots into the composite container.

## 4.7.2 Site Selection

Where applicable, wastewater samples should be collected at the location specified in the NPDES permit (if the source has such a permit). In some instances the sampling location specified in the permit, or the location chosen by the permittee, may not be acceptable for the collection of a representative wastewater sample. In such instances, the project leader or field investigator is not limited by permit specifications from collecting a sample at a more representative location. When a conflict exists between the permittee and the regulatory agency regarding the most representative sampling location, both sites should be sampled, and the reason for the conflict should be noted in the inspection or study report and field notes. Recommendations and reasons for a change in sampling location should be given to the appropriate permitting authority.

4.7.2.1 <u>Influent</u> -- Influent wastewaters are preferably sampled at points of highly turbulent flow in order to ensure good mixing; however, in many instances the most desirable location is not accessible. Preferable influent wastewater sampling points include: (1) the upflow siphon following a comminutor (in absence of grit chamber); (2) the upflow distribution box following pumping from main plant wet well; (3) aerated grit chamber; (4) flume throat; or (5) pump wet well when the pump is operating. When possible, raw wastewater samples

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should be collected upstream from sidestream returns.

- 4.7.2.2 <u>Effluent</u> -- Effluent samples should be collected at the site specified in the permit, or if no site is specified in the permit, at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters. If a conflict exists between the permittee and inspector regarding the source being sampled or the location of the most representative site, follow the procedure outlined in Section 4.7.2.
- 4.7.2.3 <u>Pond and Lagoon Sampling</u> -- Generally, composite effluent wastewater samples should be collected from ponds and lagoons. Even if the ponds or lagoons have long retention times, composite sampling is necessary because of the tendency of ponds and lagoons to have flow paths that short circuit the detention time. However, if dye studies or facility data indicate a homogeneous discharge, a grab sample may be taken as representative of the waste stream.

## 4.7.3 Sampling Techniques - General

Sampling and flow measuring are mutually dependent for a complete evaluation of a wastewater treatment plant. The investigator must know the wastewater flow variability before a sampling program can be initiated.

The choice of a flow proportional or time composite sampling scheme depends on the variability of the wastewater flow or concentration, equipment availability, and sampling location. If an investigator knows or suspects that there is significant variability in the wastewater flow or if the investigator knows nothing about the facility, a flow proportional sample should be collected. Otherwise a time composite sample would be acceptable.

Prior to sampling, the flow measuring system (primary flow device, totalizer, and recorder) should be examined (methods described in Section 5). If the flow measuring system is unacceptable, the investigator may have to install flow measurement instrumentation (Section 5). If the flow measuring system is acceptable, samples can be collected by the appropriate method.

Time composite samples collected by the Branch are based on a constant time interval between samples. A time composite sample can be collected either manually or with an automatic sampler.

Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. Refer to Appendix E for procedures describing the first method and to the NPDES Compliance Inspection Manual (2) for details on the latter two methods.

## 4.7.4 Use of Automatic Samplers

4.7.4.1 <u>General</u> -- Automatic samplers may be used when several points are to be sampled at frequent intervals or when a continuous sample is required. Automatic samplers used by the Branch must meet the following requirements:

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- Sampling equipment must be properly cleaned to avoid crosscontamination which could result from prior use (see Appendix B for cleaning procedures).
- No plastic or metal parts of the sampler shall come in contact with the water or wastewater stream when parameters to be analyzed could be impacted by these materials (See Section 4.7.6).
- The automatic sampler must be able to provide adequate refrigeration during the sampling period. This can be accomplished in the field by using ice.
- The automatic sampler must be able to collect a large enough sample for all parameter analyses (see Appendix A for required sample volumes).
- A minimum of 100 mls should be collected each time the sampler is activated if a peristaltic pump is used.
- The automatic sampler should provide a lift of at least 20 feet and the sampler should be adjustable so that the volume is not be a function of the pumping head.
- Pumping velocity must be adequate to transport solids and not allow solids to settle.
- The intake line leading to the pump must be purged before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- An adequate power source should be available to operate the sampler for 48 hours at a 30-minute sampling interval.
- Facility electrical outlets may be used if available.
- Facility automatic samplers may be used for conventional parameters if they meets SOP criteria.

Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers presently used by the Branch are included in the respective operating manuals and are not presented here.

## 4.7.4.2 Equipment Installation

4.7.4.2.1 <u>Conventional Sampling</u> -- Automatic samplers may be used to collect time composite or flow proportional samples. In the flow proportional mode, the samplers are activated by a compatible flow meter. Flow proportional samples can also be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow proportional amounts (2).

All new tubing (Dow Corning Medical Grade Silastic, or equal, in the pump

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and either Teflon® or Tygon®, or equal, in the sample train) will be used for each sampler installation.

Installation procedures include cutting the proper length of tubing, positioning it in the wastewater stream, and sampler programming.

For a time composite sample, the sampler should be programmed to collect 200 mls at 30-minute intervals or 100 mls at 15-minute intervals into a refrigerated 3-gallon glass jug. For a 5-gallon compositing container, the volume should be increased

For a flow proportional sample, the sampler should be programmed to collect a minimum of 100 mls for each sample interval, with the interval predetermined based on the flow of the waste stream.

At the end of each 24-hour sampling period, the contents of the glass compositing container with the sample should be stirred and siphoned (poured if there are no visible solids) into the respective containers, followed by immediate preservation, if required.

- 4.7.4.3 <u>Automatic Sampler Security</u> -- EPA owned equipment is assumed to be in the custody of the permittee as long as it is on their property. This assumption should be made known to permittees whenever equipment is left unattended. A lock or seal may be placed on the sampler to detect tampering. However, this does not prevent tampering with the sample collection tubing. If necessary, seals may be placed on the sampling pole and tubing line to further reduce tampering possibilities.
- 4.7.4.4 <u>Automatic Sampler Maintenance</u>. <u>Calibration</u>. <u>and Quality Control</u> To insure proper operation of automatic samplers, and thus the collection of representative samples, the procedures outlined in this section shall be used to maintain and calibrate Branch automatic samplers.

The following maintenance and calibration procedures shall be used; any differences from those prescribed procedures will be documented.

Prior to each field trip, the sampler operation will be checked by warehouse personnel. This includes operation (forward, reverse, automatic) through three cycles of purge-pump-purge; checking desiccant and replacing if necessary; checking 12-volt batteries to be used with the sampler; and repairing any item if necessary.

During each field trip, prior to initiating sampling, the purge-pump-purge cycle shall be checked at least once. The constant pumping volume using a graduated cylinder or other calibrated container will be checked at least twice, and the flow pacer that activates the sampler shall be checked to be sure it operates properly.

Upon return from a field trip, the sampler shall be examined for holes, dents, etc., and repaired, if necessary. The desiccant will be checked and replaced if appropriate. The operation (forward, reverse, automatic, etc.) will be checked and any required repairs will be made and documented. The sampler

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will then be cleaned as outlined in Appendix B.

The automatic sampler's pumping rate at multiple heads, the timing mechanisms, the multiplexer, or the flow pacer will be checked against the manufacturer's specifications and documented whenever one or more of these functions appears to be operating improperly.

Automatic sampler blanks must be collected for organic compounds and metals analyses when collecting samples for trace organic compounds analyses as described in section 4.7.6.1.

## 4.7.5 Manual Sampling

Manual sampling is normally used for collecting grab samples and/or for immediate in-situ field analyses. However, it can also be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual waste stream conditions.

The best method to manually collect a sample is to use the actual sample container which will be used to transport the sample to the laboratory. This eliminates the possibility of contaminating the sample with an intermediate collection container. The actual sample container must always be used for collecting samples for oil and grease and bacterial analyses.

A separate collection container can be used to collect the sample, from which the sample can be redistributed to other containers. If this is done, however, the container used to collect the sample must be properly cleaned (Appendix B) and must be made of a material that meets the requirements of the parameter(s) being investigated. Also, a separate container will be used at each sampling station. This will prevent cross contamination between sampling station.

If the water or wastewater stream cannot be physically reached by the sampling personnel or it is not safe to reach for the sample, an intermediate collection container may be used. The sample is collected by lowering a properly cleaned Teflon®, plastic, glass, or stainless steel collection vessel (type of collection vessel used depends on the parameter being investigated) into the material to be sampled. The collection vessel may be lowered by hand or attached to a pole or rope and then lowered into the stream.

In some cases it may be best to use a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream. If a pump is used, it is imperative that all components of the pump that come in contact with the liquid are properly cleaned (Appendix B) to insure the integrity of the sample.

Samples are manually collected by tipping the collection container in the water or wastewater stream so the mouth of the container faces upstream. The container should be rinsed with this procedure at least twice before the sample is collected (exceptions to this rinsing procedure exist if preservatives are present in the sampling container and for certain analyses such as oil and grease and bacteria).

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#### 4.7.6 Special Sample Collection Procedures

4.7.6.1 <u>Trace Organic Compounds and Metals</u> -- Trace organic compounds and metals detection limits are usually in the parts per billion range, so extreme care must be exercised to insure sample integrity.

All containers, composite bottles, tubing, etc., used in sample collection for trace organic compounds and metals analyses should be prepared as described in Appendix B.

When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This should be a Teflon®, glass, or stainless steel vessel on a pole or rope or Teflon® tubing via a peristaltic type pump and a Teflon® vacuum container attachment which converts a sample container into a vacuum container. The device which is used should be cleaned as described in Appendix B.

Personnel handling the sample should always wear a new pair of disposable surgical gloves with each set of samples collected and change as often as needed. This is necessary to prevent cross-contamination. A more detailed discussion is given in Section 4.2.10 under special precautions for trace contaminant sampling.

When an <u>automatic sampler</u> is used for collecting samples for trace organic compounds and metals analyses, the installation procedures include cutting the proper length of new Teflon® tubing, water rinsing of the entire collection system with a minimum of two gallons of Milli-Q® water, and blank collection for organics and metals. In the Milli-Q® water rinse, approximately one-half gallon is initially pumped into the composite sampler container and discarded. An additional one and one-half gallons are then pumped into the composite sampler container for distribution into the appropriate blank container. Nitric acid must be added to the metals blank container. Finally the collection tubing should then be positioned in the wastewater stream and sampler programming initiated.

4.7.6.2 <u>Bacterial</u> -- Samples for bacterial analysis will always be collected directly into the prepared glass or plastic sample bottle. The sample bottle should be kept unopened until it is to be filled. When the cap is removed, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should be held near the base and filled to within about one inch of the top without rinsing and recapped immediately. The bottle should be plunged, neck downward, below the surface and turned until the neck points slightly upward. The mouth should be directed toward the current (4, 5). Appendix A presents preservation procedures and holding times.

When the sample bottle must be lowered into the waste stream, either because of safety or impracticality (manhole, slippery effluent area, etc.), care must be taken to avoid contamination.

4.7.6.3 <u>Immiscible Liquids/Oil and Grease</u> -- Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative sample for

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Jil and grease analysis, the inspector must carefully evaluate the location of the sampling point. The most desirable sampling location is the point of greatest mixing. Quiescent areas should be avoided. Because losses of oil and grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

4.7.6.4 <u>Purseable Organic Compounds Analyses (VOA)</u> -- Samples to be analyzed for purgeable organic compounds should be collected in 40-ml septum vials with screw caps with a Teflon® lined silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the sampling program.

When sampling for purgeable organic compounds, triplicate samples should always be collected from each location. The investigator should determine if the water to be sampled contains chlorine. If the water contains no chlorine, three 40-ml vials containing four drops of concentrated HCl should be filled with the sample. The samples may be held up to 14 days before analysis. When preservation (HCl) is not feasible, samples can be held up to 7 days before analysis for purgeable aromatic hydrocarbons and 14 days for purgeable hydrocarbons compounds.

If the water contains chlorine, the following sampling and preservation procedure should be followed:

Fill an 8-ounce VOA sampling container with 8 drops of a 25% ascorbic acid solution and the water sample. Cap and mix thoroughly but gently by swirling to eliminate residual chlorine. Transfer the sample to three 40-ml VOA vials containing four drops of concentrated HC1*. Label each 40-ml vial with "P" (preserved/ascorbic acid).

The ascorbic acid and acid preservatives must be added in this order and in two separate steps.

The purgeable organics vials (40-ml) should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus." The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles; if any are present, repeat the procedure using a new 40-ml vial.

Sampling containers with preservatives should be prelabeled (i.e., P) prior to any field activities. This will reduce the chances of confusion during sampling activities by the investigation team. Sample preservation, containers, holding times, and sample volumes are listed in Appendix A.

## 4.7.7 Special Process Control Samples and Tests

Special process control samples collected and field tests performed on

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these samples are given in Appendix H.

## 4.7.8 Auxiliary Data Collection

While conducting wastewater sampling, the following information will also be obtained (if applicable):

- Field measurements pH, DO, conductivity, temperature (see Section 6 and Appendix D for standard field analytical techniques);
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples (Section 5);
- Diagrams and/or written descriptions of the wastewater treatment systems (if available);
- Photographs of pertinent wastewater associated equipment, such as flow measuring devices, treatment units, etc. (keep photolog as specified in section 3.2.3);
- Process control information on the wastewater treatment process (if applicable); and
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., will be entered in bound field logbooks or attached thereto (where applicable as specified in Section 3.5).

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#### 4.8 SURFACE WATER AND SEDIMENT SAMPLING

## 4.8.1 General

Selection of a surface water sampling location for water quality studies is based on many factors, including: study objective, water use, point source discharges, nonpoint source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, presence of structures (weirs, dams, etc.), accessibility, personnel available, etc. When sampling in estuarine systems, tidal effects must be considered in selecting sampling locations.

If the study objective is to investigate a specific location as a source of water supply, recreation, or other discrete use, then considerations such as accessibility, flow, velocity, physical characteristics, etc., are not as critical from a water quality investigation standpoint.

If the objective of a water quality study is to determine patterns of pollution, provide data for mathematical modeling purposes, conduct assimilative capacity studies, etc., where more than a single sampling location is to be investigated, then several factors become interrelated and need to be considered in sampling location selection. An excellent guide to conducting surface water stream studies is F. W. Kittrells, "A Practical Guide to Water Quality Studies" (6).

#### 4.8.2 Sample Site Selection

Before any sampling is conducted, an initial reconnaissance should be made to locate suitable sampling locations. Bridges and piers are normally good choices as sites since they provide ready access and permit water sampling at any point across the width of the water body. However, these structures may alter the nature of water flow and thus influence sediment deposition or scouring. Additionally, bridges and piers are not always located in desirable locations with reference to waste sources, tributaries, etc. Wading for water samples in lakes, ponds, and slow-moving rivers and streams must be done with caution since bottom deposits are easily disturbed, thereby resulting in increased sediment in the overlying water column. On the other hand, wadeable areas may be best for sediment sampling. In slow-moving or deep water, a boat is usually required for sampling.

Sampling station locations can be chosen without regard to other means of access if the stream is navigable by boat, especially in estuarine systems where boats frequently provide the only access to critical sampling locations.

Wading to collect samples may be feasible in some cases, but this method usually will be chosen for only very small streams. A field investigator usually has to carry a considerable amount of sampling equipment, field analytical instruments, and water samples. When it also is necessary to wear rubber boots to walk the streambed or to wade out to the main current, the physical effort involved often makes this method too difficult to be practical.

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Fresh water environments are commonly separated into two types: (1) rivers, streams, and creeks; and (2) lakes, ponds, and impoundments. Since these waterways differ considerably in general characteristics, site selection must be adapted to each. Estuarine environments are a special case and are discussed separately.

4.8.2.1 Rivers. Streams, and Creeks -- In the selection of a surface water sampling site on rivers, streams, and creeks, areas that exhibit the greatest degree of cross-sectional homogeneity shall be located. When available, previously collected data may indicate if potential sampling locations are well mixed or vertically or horizontally stratified. Since mixing is principally governed by turbulence and water velocity, the selection of a site immediately downstream of a riffle area will insure good vertical mixing. These locations are also likely areas for deposition of sediment since the greatest deposition occurs where stream velocity slows down. Horizontal (cross-channel) mixing occurs in constrictions in the channel, but because of velocity increases, the stream bottom may be scoured, and therefore, a constriction is a poor sediment sample location. In the absence of turbulent areas, the selection of a site that is clear of immediate point sources, such as tributaries and industrial and municipal effluents, is preferred for the collection of water samples.

Typical sediment depositional areas are located inside of river bends, downstream of islands, and downstream of obstructions in the water. Sites that are located immediately upstream or downstream from the confluence of two streams or rivers should generally be avoided since flows from two tributaries may not immediately mix, and at times due to possible backflow can upset the normal flow patterns.

When several stations along a stream reach are to be sampled, they should be strategically located:

- They shall be spaced at intervals based on time-of-water-travel, not distance. For example, sampling stations may be located about one-half day time-of-water-travel for the first three days downstream of a waste source (the first six stations) and then approximately one day through the remaining distance.
- If the study is to be compared to a previous study, the same sampling stations should be used, if possible, for comparison purposes.
- A station should be located whenever a marked physical change occurs in the stream channel. Example: A stream reach between two adjacent stations should not include both a long rapids section of swift shallow water with a rocky bottom, and a long section of deep, slow-moving water with a muddy bottom. Stations at each end of the combined reach would yield data on certain rates of change, such as reaeration, that would be an unrealistic average of two widely different rates. Much more would be learned of the actual natural characteristics of the stream by inserting a third sampling station within the reach, between the rapids and the quiet water sections.

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• Station locations should be selected to isolate major discharges as well as major tributaries.

Dams and weirs cause changes in physical characteristics of a stream. They usually create quiet, deep pools in river reaches that, historically were swift and shallow. Such impoundments should be bracketed. When times of water travel through them are long, stations should be established within the impoundments.

Some stream structures, such as dams, permit overflow that accomplishes significant reaeration of oxygen deficient water. In such cases, stations should be located short distances upstream and downstream from the structures to measure the rapid, artificial increase in dissolved oxygen (DO), which is not representative of natural reaeration.

A minimum of three stations located between any two points of major change in a stream is a desirable precaution, when feasible, even when the time-of-travel between the points of change is short. Major changes may consist of a waste discharge, a tributary inflow, or a significant difference in channel characteristics. The use of three stations is especially important when rates of change of unstable constituents are being determined. If results from one of only two stations in a subreach are in error for some unforeseen reason, it may not be possible to judge which of the two sets of results indicate the actual rate of change. Results from at least two of three stations, on the other hand, will probably support each other and indicate the true pattern of water quality in the subreach.

If there is interest in the effects of certain discharges or tributary streams on ambient water quality, sites should be located both upstream and downstream from the tributaries or discharges.

Unless a stream is extremely turbulent, it is nearly impossible to measure the effect of a waste discharge or tributary immediately downstream of the source. Inflow frequently "hugs" the stream bank with very little horizontal (cross-channel) mixing for some distance. This is a major consideration in estuarine environments. Samples from quarter points may miss the wastes altogether and reflect only the quality of water above the waste source. Samples taken directly in the portion of the cross section containing the wastes would indicate excessive effects of the wastes with respect to the river as a whole.

Tributaries should be sampled as near the mouth as is feasible. This may be a bridge some distance upstream from the mouth. Frequently, the mouths of tributaries may be entered from the main stream for sampling by boat. Care should be exercised to avoid collecting water from the main stream that may flow into the mouth of the tributary on either the surface or bottom because of differences in density resulting from temperature, dissolved salts, or turbidity.

Actual sampling locations will vary with the size of the water body and the amount of turbulence in the stream or river. Generally, with small streams less than 20 feet wide, a sampling site can be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth at the center of the channel is adequate to represent the entire cross-section. A sediment sample can

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also be collected at the center of the channel. For slightly larger streams, at least one vertical composite should be taken from mid-stream, with samples taken just below the surface, at mid-depth, and just above the bottom. Of course, DO, pH, temperature, conductivity, etc. should be measured on each aliquot of the vertical composite. For large rivers, several locations across the channel width should be sampled. Vertical composites across the channel width should be located in a manner that is roughly proportional to flow, i.e., they should be closer together toward mid-channel, where most of the flow travels, than toward the banks, where the proportion of total flow is smaller. The number of vertical composites required and the number of depths sampled for each are usually determined in the field by the sampling crew. This determination is based on a reasonable balance between the following two considerations.

- 1. The larger the number of subsamples, the more nearly the composite sample will represent the water body.
- 2. Taking subsamples is time-consuming and expensive, and increases the chance of contamination.

In most circumstances, a number of sediment samples should be collected along a cross-section of a river or stream in order to adequately characterize the bed material. A common procedure is to sample at quarter points along the cross-section of the site selected. When the sampling technique or equipment requires that the samples be extruded or transferred at the site, they can be combined into a single composite sample. However, samples of dissimilar composition should not be combined but should be stored for separate analysis in the laboratory. To insure representative samples, the preferred method is diver deployed coring tubes.

4.8.2.2 <u>Lakes. Ponds. and Impoundments</u> -- Lakes, ponds, and impoundments have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. Occasionally, an extreme turbidity difference may occur vertically where a highly turbid river enters a lake, and each layer of the stratified water column needs to be considered. Since the stratification is caused by water temperature differences, the cooler, heavier river water is beneath the warmer lake water. A temperature profile of the water column as well as visual observation of lake samples can detect the different layers and they can be sampled separately.

The number of water sampling sites on a lake, pond, or impoundment will vary with the purpose of the investigation as well as the size and shape of the basin. In ponds and small impoundments, a single vertical composite at the deepest point may be sufficient. Similarly, the measurement of DO, pH, temperature, etc., is to be conducted on each vertical composite aliquot. In naturally-formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam.

In lakes and larger impoundments, several vertical subsamples should be composited to form a single sample. These vertical sampling locations are often taken along a transect or grid. Again, the number of vertical subsamples and the depths at which subsamples are taken are usually at the discretion of the

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sampling crew. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones, but normally a composite shall consist of several vertical subsamples collected at various depths.

In lakes with irregular shape and with several bays and coves that are protected from the wind, additional separate composite samples may be needed to adequately represent water quality. Similarly, additional samples should be taken where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

When collecting sediment samples in lakes, pond, and reservoirs, the site selected should be approximately at the center of water mass. This is particularly true for reservoirs that are formed by the impoundment of rivers or streams. Generally, the coarser grained sediments are deposited near the headwaters of the reservoir, and the bed sediments near the center of the water mass will be composed of fine-grained materials. The shape, inflow pattern, bathymetry, and circulation must be considered when selecting sediment sampling sites in lakes or reservoirs.

- 4.8.2.3 <u>Estuarine Waters</u> -- Estuarine areas are zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Estuaries are generally categorized into three types, dependent upon freshwater inflow and mixing properties (7, 8, 9, and 10):
  - Mixed estuary -- Characterized by an absence of vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically this type of estuary is found in major freshwater sheetflow areas, featuring shallow depths.
  - Salt wedge estuary -- Characterized by a sharp vertical increase in salinity and channelized freshwater inflow into a deep estuary. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally, back and forth, with the tidal phase.
  - Oceanic estuary -- Characterized by salinities approaching full strength oceanic waters. Seasonally, freshwater inflow is small with the preponderance of the fresh saline water mixing occurring near, or at, the vegetated shore line.

A reconnaissance investigation should be conducted for each estuarine study unless prior knowledge of the estuarine type is available. The reconnaissance should focus upon the freshwater and oceanic water dynamics with respect to the study objective. In this regard, National Oceanic Atmospheric Administration tide tables and USGS freshwater surface water flow records provide perspective to the estuary dynamics. The basic in-situ measurement tools for reconnaissance are: a boat, recording fathometer, salinometer, and dissolved oxygen meter. These instruments coupled with the study objective or pollution source location, whether it is a point or nonpoint source problem, provide the focus for setting

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sampling locations. More often than not, preplanned sampling locations in estuarine areas are changed during the actual study period. Because of the dynamics of estuaries, initial sampling often reveals that the study objective could be better served by relocating, adding, or deleting sampling locations.

Sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides (11).

All estuarine sampling programs conducted by Branch personnel shall include vertical salinity measurements at one- to five-foot increments coupled with vertical DO and temperature profiles. A variety of water sampling devices are used, but in general, the Van Dorn (or similar type) horizontal sampler is employed.

Samples are normally collected at mid-depth in areas where the depths are less than 10 feet, unless the salinity profile indicates the presence of a halocline (salinity stratification). In that case, samples are collected from each stratum. Depending upon the study objective, when depths are greater than 10 feet, water samples may be collected at the one-foot depth, mid-depth, and one-foot from the bottom.

In general, estuarine investigations are two phased, with study investigations conducted during wet and dry periods. Depending upon the freshwater inflow sources, estuarine water quality dynamics cannot normally be determined by  $\alpha$  single season study.

For a complete discussion of estuarine systems, refer to references 7, 8, 9, and 10.

4.8.2.4 <u>Control Stations</u> -- In order to have a basis of comparison of water quality, the collection of samples from control stations is always necessary. A control station above the source of waste is fully as important as are stations below, and should be chosen with equal care to ensure representative results. At times it may be desirable to locate two or three stations above the waste inflow to establish the rate at which the unstable material is changing. The time of travel between the stations should be sufficient to permit accurate measurement of the change in the constituent under consideration.

## 4.8.3 Sampling Equipment and Techniques

- 4.8.3.1 <u>General</u> -- Any equipment or sampling techniques used to collect a sample are acceptable as long as they do not cause the integrity of the sample to be violated and which provide a sample which is representative of the stream being sampled.
- 4.8.3.2 <u>Water Sampling Equipment/Techniques</u> -- The physical location of the investigator when collecting a sample may dictate the equipment to be used. If surface water samples are required, direct dipping of the sample container into the stream is desirable. This is possible, however, only from a small boat, a pier, etc., or by wading in the stream. Wading, however, may cause bottom deposits to rise and bias the sample. Wading is acceptable if the stream has a noticeable current (is not impounded), and the samples are collected directly

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into the bottle while pointed upstream. If the stream is too deep to wade or if the sample must be collected from more than one water depth or from a bridge, etc., supplemental sampling equipment must be used.

Teflor bailers may be used for surface water sampling, if the data requirements do not necessitate a sample from a strictly discrete interval of the water column. A closed top bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water column, water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful where strong currents are found, or where a discrete sample at a specific depth is required.

If discrete samples are desired at a specific depth, and the parameters to be measured do not require a Teflon® coated sampler, a standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position to allow free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill respective sample bottles. With a rubber tube attached to the valve, DO sample bottles can be properly filled by allowing an overflow. With multiple depth samples, care should be taken not to stir up the bottom sediment and thus bias the sample.

A plastic bucket can be used to collect samples if the parameters to be analyzed do not preclude it. However, the bucket should be rinsed twice with the sample water prior to collection of the sample. All field equipment should follow standard cleaning procedures.

- 4.8.3.3 <u>Sediment Sampling Equipment/Techniques</u> -- To collect a sediment sample from a streambed, a variety of methods can be used. Dredging (Peterson, Eckman, Ponar), coring, and scooping (BMH-60) are available. Regardless of the method used, precautions shall be taken to insure that the sample collected is representative of the streambed. These methods are discussed in the following paragraphs.
- 4.8.3.3.1 <u>Dredging</u> -- For routine analyses, the Peterson dredge can be used when the bottom is rocky, in very deep water, or when the stream velocity is high. The dredge should be lowered very slowly as it approaches bottom, because it can displace and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high velocities. It should not be used from a bridge more than a few feet above the water, because the spring mechanism which activates the sampler can be damaged by the messenger if dropped from too great a height.

Tack man greeky!

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Roral

The Ponar dredge is a modification of the Peterson dredge and is similar in size and weight. It has been modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

4.8.3.3.2 <u>Corers</u> -- Core samplers are used to sample vertical columns of sediment. They are particularly useful when a historical approach to sediment deposition is desired for they preserve the sequential layering of the deposit. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand push tubes to weight or gravity driven devices.

Coring devices are particularly useful in pollutant monitoring because the "shock wave" created by descent is minimal, thus the fines of the sediment-water interface are only minimally disturbed; the sample is withdrawn intact permitting the removal of only those layers of interest; core liners manufactured of glass or Teflon® can be purchased, thus reducing possible sample contamination; and the samples are easily delivered to the lab for analysis in the tube in which they were collected. The disadvantage of coring devices is that a relatively small surface area and sample size is obtained often necessitating repetitive sampling in order to obtain the required amount for analysis. Because it is felt that this disadvantage is offset by the advantages, coring devices are recommended in sampling sediments for trace organic compounds or metals analyses.

In shallow, wadeable waters, the direct use of a core liner or tube manufactured of Teflon or glass is recommended for the collection of sediment samples. Their use can also be extended to deep waters when SCUBA equipment is Teflone is preferred to avoid glass breakage and possible sample available. loss. Stainless steel push tubes are also acceptable and provide a better cutting edge and higher strength than Teflon®. The use of the glass or Teflon® tube by itself eliminates any possible metal contamination from core barrels, cutting heads, and retainers. The tube should be approximately 12 inches if only recently deposited sediments (8 inches or less) are to be sampled. Longer tubes should be used when the depth of the substrate exceeds eight inches. Soft or semiconsolilated sediments such as mud and clays have a greater adherence to the inside of the tube and thus can be sampled with larger diameter tubes. Because coarse or unconsolidated sediments such as sands and gravel tend to fall out of the tube, a small diameter is required for them. A tube about two inches in diameter is usually the best size. The wall thickness of the tube should be about 1/3 inch for either Teflon® or glass. The inside wall may be filed down at the bottom of the tube to facilitate entry of the liner into the substrate.

Caution should be exercised not to disturb the area to be sampled when the sample is obtained by wading in shallow water. The core tube is pushed into the substrate until only four inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is pushed will facilitate greater penetration and cut down on core

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compaction. The tube is then capped with a Teflon® plug or a sheet of Teflon® held in place by a rubber stopper or cork. After capping, the tube is slowly extracted, the negative pressure and adherence of the sediment keeping the sample in the tube. Before pulling the bottom part of the core above the water surface, it too is capped.

For a detailed description of several types of coring devices, refer to "Sampling Protocols for Analysis of Toxic Pollutants in Ambient Water, Bed Sediment and Fish" (12).

4.8.3.3.3 <u>Scooping</u> -- If the water is wadeable, the easiest way to collect a sediment sample is to scoop the sediment using a stainless steel spoon or scoop. This reduces the potential for cross-contamination. This can be accomplished by wading into the stream, and while facing upstream (into the current), scooping the sample along the stream bottom in the upstream direction. If the stream is too deep to wade but less than eight feet deep, a stainless steel scoop attached to a piece of conduit can be used either from the banks if the stream is narrow or from a boat.

If the stream has a significant flow and is too deep to wade, a BMH-60 sampler may be used. It is not particularly efficient in mud or other soft substrates because its weight will cause penetration to deeper sediments, which are not desired when sampling for priority pollutants. It is also difficult to release secured samples in an undisturbed fashion that would readily permit subsampling. The BMH-60 may be used for priority pollutant sampling provided that caution is exercised by only taking subsamples that have not been in contact with the metal walls of the sampler.

4.8.3.3.4 <u>Mixing</u> -- Regardless of the method of collection, sediment samples collected for chemical analysis should be thoroughly mixed (except for purgeable organic compounds analysis) before being placed in the appropriate sample containers, as specified in Section 4.2.10.

# 4.8.4 Special Sample Collection Techniques

4.8.4.1 Trace Organic Compounds and Metals -- Since trace organic compounds and metals are usually found in extremely low concentrations in ambient waters, the possibility of contamination is greater than with sediment or fish. Precautions must be taken with sampling equipment and preservatives to ensure that contamination does not occur.

Direct dipping of the sample containers is the preferred method of collecting surface water sample for trace levels pollutants analyses. If samples are to be split for enforcement or quality control purposes, and duplicate samples will not suffice, a sufficient volume for all sample containers should be collected in a large glass compositing container and then, with mixing, be alternately siphoned or poured into the respective sample bottles. This technique is not to be used for samples collected for purgeable organic compounds analysis.

Many contaminants are partitioned more strongly to sediments than water;

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thus, if these contaminants have been deposited recently and are not quickly degraded or desorbed, they are evident in the sediment analysis. Ideally, only Teflon®, stainless steel, or glass should be used in sediment sampling for trace level contaminant analyses. The method using glass or Teflon® coring tubes, was discussed previously, is the preferred technique.

In many situations when the water is deep, divers are not available, or the only sampling location is from a bridge, a dredge may be used. In these cases, a high grade stainless steel Ponar dredge (properly cleaned, Appendix B) shall be used to collect the sediment samples. Direct scooping of the streambed sediment as described earlier is acceptable.

4.8.4.2 <u>Bacterial</u> -- Samples for bacteriological examination must be collected in bottles properly sterilized and protected against contamination. The preferable method is to scoop up the water with the open bottle just below the surface. This method usually is used when sampling by boat. While the bottle is open, both bottle and stopper must be protected against contamination. A small amount of water should be poured from the bottle after filling to leave an air space for subsequent shaking in the laboratory. The bottle should be closed at once.

When sampling from a bridge, the sterilized sample bottle can be placed in a weighted frame, opened, and then lowered to the water with a string or rope. The bottle can also be lowered by attaching twine directly to it. Care should be taken not to dislodge dirt or other material from the bridge that will fall into the open bottle. The mouth of the bottle may be faced upstream by swinging the bottle downstream under the bridge and dropping it quickly but without excessive slack in the rope. The bottle is then pulled upstream and out of the water.

Special equipment for collecting sterile samples at various depths is available. The JZ sampler, for example, includes a metal frame to hold the sample bottle, two sterile glass tubes connected by a rubber tube and inserted through the sterile bottle stopper, and a messenger. One of the glass tubes is bent so that the upper portion is horizontal. This portion is positioned next to a rope. The messenger breaks the tube, which allows the bottle to fill. The bottle, glass tube and rubber tube must be properly sterilized before use. Additional guidance for collecting samples for bacterial analyses is given in References 4 and 5.

# 4.8.5 Auxiliary Data Collection

A bound field log book will be used to record daily activities, describe sampling locations and techniques, list photographs taken, etc., as specified in Section 3.5. Visual observations are particularly significant and may prove invaluable in interpreting water quality study results. These visual observations, including weather and stream conditions (including tide stage) during the investigation should also be recorded in field records.

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# 4.8.6 Specific Sampling Equipment Quality Assurance Procedures

All water and sediment sampling equipment shall be cleaned as outlined in Appendix B after field use and before being stored. This equipment shall be inspected and tested before being issued for each field study. Any repairs necessary shall be made and recorded.

During field studies, this equipment shall be cleaned as outlined in Appendix B and all such cleaning procedures shall be documented in field records.

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#### 4.9 GROUND WATER SAMPLING

#### 4.9.1 General

Ground-water sampling may be required for a variety of reasons, such as examining potable or industrial water supplies, checking for and/or tracking contaminant plume movement in the vicinity of a land disposal or spill site, RCRA compliance monitoring, or examining a site where historical information is minimal or non-existent but where it is thought ground water contamination could have occurred.

Ground water is usually sampled through an in-place well, either temporarily or permanently installed. However, it can also be sampled anywhere ground water is present, as in a pit or a dug or drilled hole.

Occasionally, a well will not be in the ideal location to obtain the sample needed (for example, to track a contaminant plume). In that case, a well will have to be installed, and it may be either a temporary or permanently installed well. An experienced and knowledgeable person will need to locate the well and supervise its installation so that the samples ultimately collected will be representative of the ground water.

The procedures covered in this section are those used by the Branch. Additional guidance is given in the "RCRA Ground Water Monitoring Technical Enforcement Guidance Document" (TEGD) (13); the ground water sampling procedures used by Branch personnel will meet or exceed the requirements of this document.

# 4.9.2 Site Selection

The relationship of the following factors to potential pollution sources shall be considered and evaluated when selecting ground-water sampling sites:

- the direction of ground-water flow, depth to ground water, thickness of the aquifer (if applicable);
- type of stratigraphy;
- presence of perched water tables;
- types of soils;
- depth to bedrock;
- type of vegetation;
- surface drainage patterns;
- type of topography;
- general land use;

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 and surface features such as rock outcrops, seeps, springs, streams, rivers, and wet areas (14).

The area of interest should be located on an aerial photograph, a USGS 7.5 minute quadrangle map, a USDA soils map, and/or any other appropriate map that shows topography and general relationships between surface features. Aerial photographs can usually be obtained at the local Agricultural Stabilization Conservation Service (ASCS) office or the local county tax office. USGS 7.5 minute quadrangle maps can be acquired from the State Geological Survey or from the USGS, and soils maps from the USDA-SCS (Soil Conservation Service). A visual inspection of the area may be sufficient to evaluate and determine the surface conditions and their relationship to the subsurface conditions (14). In some cases, surface conditions and subsurface conditions cannot be correlated by site inspection or reconnaissance. When this occurs, a more detailed study, possibly involving test drilling, will have to be conducted.

It is extremely important to sample the unconfined or surficial aquifer downgradient of potential pollution sources or spills to determine if it (the most easily contaminated aquifer) has been affected. Generally the direction of ground-water flow can be estimated by two vectors - one in the direction of surface water flow (i.e., downstream) and another toward the nearest surface water stream or river, if present. The relative magnitude of these vectors will vary according to site conditions and in some instances both direction and magnitude may be changed by construction activities. If both a shallow and a deep aquifer are involved in the zone of interest, a screening study will reveal whether or not the deep aquifer should be sampled and a more detailed study is required. To adequately assess subsurface conditions, a minimum of three wells is required; one in the upgradient portion of the area of interest, one in the middle portion, and one in the downgradient portion. In some cases, a more complex system of wells may be needed to define the subsurface conditions, especially in establishing the depth to the shallow ground-water aquifer and the direction of ground-water movement. Site conditions and the scope of the project will determine the total number of wells required. Existing wells should be used when possible. Where permanent well installation is necessary, the wells should be installed according to the procedures in Appendix E.

# 4.9.3 Purging Equipment and Techniques

4.9.3.1 General -- Wells shall be purged before taking samples in order to clear the well of stagnant water which is not representative of aquifer conditions. The method of purging is to pump the well until three to five times the volume of standing water in the well has been removed and until the specific conductance, temperature, and pH of the ground water stabilizes. Normally, a combination of the two methods is employed (i.e., specific conductance, temperature, and pH are measured at intervals and three to five volumes are purged). If a well is pumped dry, this constitutes an adequate purge and the well can be sampled following recovery (15, 17). However, if possible, monitoring wells should not be pumped dry. If the well is pumped dry, water that has been trapped in the sandpack may be sampled. In addition, as water re-enters the well it may cascade down the well screen and strip volatile contaminants.

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4.9.3.2 Equipment Available -- Monitoring well purging is accomplished by using in-place plumbing/pumps or when in-place pumps are not available, by using EPA equipment; either a peristaltic, turbine, bladder, centrifugal, or other appropriate pump, depending on well depth. A Teflone, closed top bailer may be used for purging; however, bailing may stir up sediment in the well if conducted improperly.

Other monitoring equipment used during purging includes water level indicators, pH meters, thermometers, and conductivity bridges (See Section 6, Field Analytical Procedures).

# 4.9.3.3 Purging Techniques (Wells Without Plumbing or In Place Pumps)

- 4.9.3.3.1 <u>General</u> -- For permanently installed wells, the depth of water shall be determined (if possible) before purging. This can be accomplished by attaching a weight on the end of a tape and lowering it into the well until it touches the water, or by use of a mechanical or electrical water level indicator (see Ground-Water Level Measurement Techniques, Section 7.7). Branch personnel shall exercise extreme caution during this procedure to prevent contamination of the ground water. This is a critical concern when samples for trace organic compounds or metals analyses are collected.
- 4.9.3.3.2 <u>Using Pumps to Purge</u> -- When suction lift or centrifugal pumps are used, only the intake line is placed into the water column. To minimize contamination, the line placed into the water is either standard cleaned (see Appendix B) Teflone, in the case of the suction lift pumps, or standard cleaned stainless steel pipe attached to a hose, when centrifugal pumps are used.

When submersible pumps (bladder, turbine, displacement, etc.) are used, the pump itself is lowered into the water column. The pump must be cleaned as specified in Appendix B.

- 4.9.3.3.3 <u>Using Bailers to Purge</u> -- Standard cleaned (Appendix B) closed-top Teflon® bailers with Teflon® leaders and new nylon rope are lowered into top of the water column, allowed to fill, and removed and then the water is discarded.
- 4.9.3.3.4 Field Care of Purging Equipment -- Regardless of which method is used for purging, new plastic sheeting shall be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they need to be placed on the ground during the purging or they accidentally come into contact with the ground surface. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool, both during transporting and during field use, to further minimize contamination from the transporting vehicle or ground surface.
- 4.9.3.3.5 <u>Purging Entire Water Column</u> -- The pump/hose assembly or bailer used in purging should be lowered into the top of the standing water column and not deep into the column. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump is placed deep into

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the water column, the water above the pump may not be removed, and the subsequent samples collected may not be representative of the ground water.

To minimize cross contamination between wells, no more than three to five feet of hose should be lowered into the water column. If the recovery rate of the well is faster than the pump rate, the pump may be left hanging at the initial level until an adequate volume has been purged. If the pump rate exceeds the recovery rate of the well, the pump will have to be lowered, as needed, to accommodate the drawdown.

After the pump is removed from the well, all wetted portions of the hose and the pump shall be cleaned as outlined in Appendix B.

Careful consideration shall be given to using pumps to purge wells which are excessively contaminated with oily compounds, because it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When these type wells are encountered, alternative purging methods, such as bailers, should be considered.

# 4.9.3.4 Purging Techniques - Wells With In Place Plumbing

4.9.3.4.1 <u>General</u> -- In-place plumbing is found at water treatment plants, industrial water supply wells, private residences, etc. The objective of purging is the same as with monitoring wells without in place pumps, i.e., to ultimately collect a sample representative of the ground water.

The volume to be purged depends on several factors: whether the pumps are running continuously or intermittently; how close to the source the sample can be collected; and the presence of any storage/pressure tanks between the sampling point and the pump. If storage/pressure tanks are present, an adequate volume must be purged to totally exchange the volume of water in the tank.

- 4.9.3.4.2 <u>Continuously Running Pumps</u> -- If the pump runs continuously, and the sample can be collected prior to a storage/pressure tank, no purge, other than opening a valve and allowing it to flush for a few minutes, is necessary.
- 4.9.5.4.3 <u>Intermittently Running Pumps</u> -- If the pump runs intermittently, it is necessary to determine the volume to be purged, including storage/pressure tanks that are located prior to the sampling location. The pump should then be run continuously until the required volume has been purged.

# 4.9.4 Sampling Equipment and Techniques

4.9.4.1 Equipment Available -- Sampling equipment used by the Branch includes closed-top Teflon® bailers and the peristaltic pump/vacuum jug assembly.

Other monitoring equipment used during sampling includes water level indicators, pH meters, thermometers, and conductivity bridges (see Sections 6 and 7).

4.9.4.2 <u>Sampling Techniques -- Wells With In Place Plumbing</u> -- Samples should be collected following purging from a valve or cold water tap as near to

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the well as possible. Samples should be collected directly into the appropriate containers (see Standard Sample Containers, Appendix A). Also, refer to the Potable Water Supply discussion in Section 4.10.

4.9.4.3 <u>Sampling Techniques -- Wells Without Plumbing</u> -- Following purging, samples should be collected using a peristaltic pump/vacuum jug procedure, if possible, or with a closed top Teflon® bailer. The pump used for purging generally should not be used for sampling. When the peristaltic pump is used, samples for purgeable organic compounds analyses should be collected using a bailer or by allowing the Teflon® tube to fill and then allowing the water to drain into the sample vials. All equipment shall be cleaned using the procedures described in Appendix B. Also, refer to the Potable Water Supply discussion, Section 4.10, for additional information.

When bailing, new plastic sheeting should be placed on the ground around each well to provide a clean working area. The nylon rope should be attached to the bailer via a Teflon® coated stainless steel wire. This coated wire is attached to the bailer semi-permanently and is decontaminated for reuse as the bailer is cleaned.

# 4.9.5 Special Sample Collection Procedures

- 4.9.5.1 Trace Organic Compounds and Metals -- Special sample handling procedures shall be instituted when trace contaminant samples are being col-All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which come into contact with the water in the well must be cleaned in accordance with the cleaning procedures described in Appendix Pumps shall not be used for sampling, unless the interior and exterior portions of the pump and discharge hoses can be thoroughly cleaned. Blanks should be collected to determine the adequacy of cleaning prior to collection of any sample using a pump. Peristaltic pumps using Teflon® tubing and a Teflon® insert can be used to collect samples without the sample coming into contact with the pump. This is accomplished by placing the Teflon® insert into the opening of a standard cleaned 4-liter glass container. The Teflon® tubing connects the container to the pump and sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without coming into contact with the pump tubing. Samples for purgeable organic compounds analyses shall be collected with well bailers. The procedures given in the General Considerations, Special Precautions for Trace Contaminant Sampling (Section 4.2.9) shall be followed.
- 4.9.5.2 <u>Filtering</u> -- As a standard Branch policy, ground-water samples will not be filtered. However, if samples are filtered, then both filtered and non-filtered samples will be submitted for analyses. Proper well installation and development (Appendix E) as well as proper well purging techniques should be utilized to minimize the turbidity of samples. If filtered samples for metals analyses must be collected, an additional unfiltered sample will also be collected for metals analyses. Samples for organic compounds analyses shall not be filtered.

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4.9.5.3 <u>Bacterial Sampling</u> -- Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in <u>Sampling for Organic Chemicals and Microorganisms in the Subsurface</u> (19) as well as References 4 and 5. Additional information on bacterial sampling is also included in Section 4.10 (Potable Water Supplies), Section 4.7 (Wastewater Sampling), and Section 4.8 (Surface Water Sampling).

#### 4.9.6 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground-water samples shall be cleaned as outlined in Appendix B and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures conducted in the field (Appendix B), or field repairs shall be thoroughly documented in field records.

#### 4.9.7 Auxiliary Data Collection

Water table measurements from the top of the well casings (referenced to National Geodetic Vertical Datum) in permanent wells, and ground surface elevations in temporary wells should be made to determine the general direction of ground-water flow and gradient. The methodology to be used to determine well water levels are given in Section 7. Tracer dyes and radioactive and thermal detection methods can be used to determine direction and velocities of flow (14). Also, a study of the general topography and drainage patterns will generally indicate direction of ground-water flow.

Water table measurements shall not be taken until the water table has stabilized, preferably 24 hours after well installation for permanent wells (20). The ground surface elevation at the wells should be determined by standard engineering survey practices as outlined in Section 7.

In addition to water level measurements, the pumping rate used to purge a well, the volume of water in wells, and drillers' logs are examples of auxiliary data that should be collected during ground-water sampling activities. This information should be documented in field records. Methodology for obtaining these data are given in the following sections.

Temperature, specific conductance, and pH shall be measured each time a well is sampled. Stabilization of these parameters is measured during the purging process to evaluate the adequacy of the purging procedure. In this situation, the final measurements for these parameters prior to sampling shall be considered the measurement of record for the well. If these parameters were not evaluated during purging, they shall be obtained prior to sampling. Methodology for obtaining these data are given in Section 6.

4.9.7.1 Well Pumping Rate - Bucket/Stop Watch Method -- The pumping rate of a pump can be determined by collecting the flow of water from the pump in a bucket of known volume and timing how long it takes to fill the bucket. The pumping rate should be in gallons per minute. This method shall be used only with pumps with a constant pump rate, such as gasoline powered or electric

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submersible pumps. It should not be used with battery powered pumps. As the batteries lose their charge, the pump rate decreases so that pumping rate calculations using initial, high pump rates are erroneously high.

4.9.7.2 <u>Volume of Water in Wells</u> -- In order to purge wells, the volume of water in the well should be known. To determine the volume, the following method should be used; measure the distance from the bottom of the well to the static water level, then measure the inside diameter of the well or casing. Obtain the volume of the well by the formula:

 $V = 0.041 d^2h$ 

Where h - depth of water in feet d - diameter of well in inches V - volume of water in gallons

If preferred, a quick reference nomograph or table may be used.

Additional ground-water related data can be obtained from most local, state, and federal agencies dealing with water resources. Some states require well drillers to be licensed, and all work performed on wells must be reported to the state on prescribed forms. These forms are available to the public, so a study of wells installed in the area of interest may provide background infornation as to the subsurface conditions. State geological surveys, as well as the JSGS, have various types of water related papers and reports on all phases of ground-water studies in each state. City and county governments usually have departments that deal with water related projects that may provide data for the local area. Federal agencies such as the SCS, U. S. Army Corps of Engineers, the Bureau of Reclamation, U. S. Forest Service, Science and Education Administration, and the U. S. Public Health Service have water programs which may provide data. Other sources include the Bureau of Mines, colleges, universities, and technical societies such as American Association of Petroleum Geologists, American Institute of Mining and Metallurgical Engineers, American Water Well Association, Association of Engineering Geologists, and Geological Society of America (14, 21).

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#### 4.10 SAMPLING OF POTABLE WATER SUPPLIES

# 4.10.1 General

When sampling potable water supplies, utmost care must be taken to insure that samples are representative of the water supply being sampled. This is important not only from a technical and public health perspective, but also from a public relations standpoint. Poor sampling techniques may result in incorrect results (either not detecting a compound which is present or by contaminating the sample and falsely indicating a compound which is not present). If incorrect results are disclosed to the public, it may be impossible to change public opinion when correct results are reported.

# 4.10.2 Sampling Site Selection/Sampling Techniques

Even though the same care and techniques used in wastewater, ground-water, etc., sampling (including thorough documentation of location, date, time, etc.) are used by Branch personnel in potable water supply sampling, there are certain additional special procedures which shall be used.

When water samples are collected from wells, either by mechanical or hand pumping, the wells must be purged before the sample is collected (see Section 4.9 for ground-water sampling methods). This procedure insures that water representative of the formation is sampled, not the standing water in the well casing, pipes, or holding tank. As a rule of thumb, at least one volume of water in the well casing and storage tank should be evacuated (a 15-minute period is usually sufficient for residential wells). After purging for about 15 minutes, the pH, conductivity, and temperature should be measured until constant values are obtained. This procedure insures that any contaminants that might have entered the area of the tap from external sources have been removed (19).

Potable water samples shall be representative of the water quality within a given segment of the distribution network. Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest and should not be separated from the segment of interest by a storage tank. The sampling tap must be protected from exterior contamination associated with being too close to the sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collecting procedure since it is difficult to place a bottle under a low tap without grazing the nack interior against the outside faucet surface. Leaking taps that allow water to flow out from around the stem of the valve handle and down the outside of the faucet, or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations. Aerator, strainer, and hose attachments on the tap must be removed before sampling. These devices can harbor a bacterial population if they are not cleaned routinely or replaced when worn or cracked. Whenever a steady stream of water cannot be obtained from taps, after such devices are removed, a more suitable tap shall be sought. Taps where the water flow is not steady should be avoided because temporary fluctuation in line pressure may cause sheets of microbial growth that are lodged in some pipe section or faucet connection to break loose. The cold water tap should be opened for two or three minutes or for sufficient time to permit clearing the service line; a smooth-flowing water stream at moderate

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pressure without splashing should be obtained. Then, without changing the water flow which could dislodge some particles in the faucet, the samples can be collected (19).

Occasionally, samples are collected to determine the contribution of transmission pipes, water coolers, water heaters, etc., to the quality of water in private residences, offices, etc. The purpose of these investigations may be to determine if metals, e.g., lead, are being dissolved into the water supply. In these cases, it may be necessary to insure that the water source has not been used for a specific time interval, e.g., over a weekend or a three- or four-day holiday period. Sample collection may consist of collecting a sample of the initial flush, collecting a sample after several minutes, and collecting another sample after the system being investigated has been completely purged.

Regardless of the type of sample bottle being used, the bottle cap should not be placed on the ground or in a pocket. Instead, hold the bottle in one hand and the cap in the other, using care not to touch the inside of the cap. Exercise care not to lose the Teflon® liner in certain bottle caps. Avoid contaminating the sample bottle with fingers or permitting the faucet to touch the inside of the bottle. When sampling for bacterial content, the bottle should not be rinsed before use. This may not only contaminate the bottle but also remove the thiosulfate dechlorinating agent (if used). When filling any container, care should be taken so splashing drops of water from the ground or sink do not enter into either the bottle or cap. In order to avoid dislodging particles in the pipe or valve, do not adjust the stream flow while sampling.

When sampling at a water treatment plant, samples should be collected both from the raw water supply and after chlorination.

Triplicate samples will always be collected for purgeable organic compounds analyses and duplicate samples for bacterial analyses. Single samples may be collected for extractable organic compounds, metals, phenol, cyanide, and conventional parameter analyses. The procedures given in Section 4.2.11 (Special Precautions for Trace Contamination Sampling) and in Section 4.7.6.4 (Purgeable Organic Compounds Sample Collection) shall always be followed when potable water supplies are sampled.

Branch personnel shall always obtain the name(s) of the resident or water supply owner/operator and the resident's exact mailing address, as well as the resident's home and work telephone numbers. The information is required so that the residents or water supply owner/operators can be informed of the results of the sampling program.

# 4.10.3 <u>Sampling Equipment/Specific Sampling Equipment Quality Assurance</u> Techniques

Sampling equipment and specific equipment quality assurance techniques are contained in Section 4.8 (Surface Water Sampling) and Section 4.9 (Groundwater Sampling).

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#### 4.11 SOIL SAMPLING

#### 4.11.1 General

Soil sampling at hazardous waste sites will typically be approached in a totally different manner than sampling of other media. Sampling locations and rationale for other media are usually easily defined. For example, ground water samples may be collected at existing monitoring wells; surface water and sediment samples are usually collected from well defined surface drainage patterns at easily rationalized locations with respect to the suspected problem; and waste sources, such as drums, tanks, and piles, are easily identified sampling targets. Occasionally, surface soils may be stained or show evidence of vegetative stress, indicating that a contaminant may be present, but in many cases there may not be any direct evidence to suggest that a particular location is a candidate for soil sampling. The sampler may, in fact, be faced with investigating a virtually invisible contaminant distribution pattern, both in the surficial material, as well as in the subsurface region.

### 4.11.2 Sampling Location/Site Selection

Areas selected for soil sampling shall be strategically located in order to collect a representative fraction of the soils with the minimum number of samples. Although it is not always feasible to conduct a site reconnaissance prior to an investigation, a site reconnaissance can eliminate many uncertainties with respect to site characteristics and result in more complete and successful soil sampling studies. A surface inspection of the subject area should be made to locate pertinent features (e.g., rock outcrops, drainage patterns, surface runoff, ponds, lakes, wat areas, seeps, springs, permanent structures, fill areas, erosional areas, depositional areas, etc.) and to evaluate the relationship between these features and potential sources of pollution. A knowledge of these relationships and conditions, particularly soil conditions (type and thickness of soil overburden) and water table conditions are extremely important in developing sampling plans.

In addition to what is normally considered soil, i.e., in situ weathered rock overburden, soil samples may also consist of what is more correctly considered sediment, which has been deposited by both overland sheet runoff, as well as flow in normally dry wet-weather swales. The location of sediment sampling locations in these types of depositional areas is a useful screening tool, providing an indication of the presence of contaminants from the larger area contributing the sediment.

Initial investigations at most sites will consist of "screening-type" studies. Sampling for these investigations will generally be confined to a small number of surface or shallow subsurface samples. Typically, samples would be collected from depositional areas within and around the periphery of the site, as well as from obviously contaminated areas. Based on the results of the initial site-screening studies, more detailed studies, involving considerably more samples and with a greater emphasis on subsurface sampling, are usually required to fully characterize soil contamination at a site.

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Study plans or work plans for soil sampling investigations must be carefully conceived with respect to the <u>study objectives</u>. This, in turn, requires a careful consideration of the types of samples to be collected, as well as the sampling methods to be employed. These areas are discussed below.

# 4.11.3 Basic Considerations for Soil Sampling

Three basic considerations, with respect to sample type, should be evaluated when developing a soil sampling plan and establishing investigation objectives. Should the samples be random, biased, or grid-based? Will they be collected from the surface or subsurface? Will a particular sample be a grab sample or a composite sample? When all of these questions are answered, it will be found that many investigations will involve the collection of most combinations of the above types of samples. A discussion of these considerations is found in the following sections.

4.11.3.1 Random. Biased and Grid-Based Sampling -- Generally, unless there is a strong indication of contamination, such as staining, or there are distinct depositional areas which provide excellent screening samples, soil samples collected for small investigations with limited areal extent, such as screening site investigations, must be <u>randomly</u> selected from several areas within the suspected area of contamination. Random in this sense is synonymous with casual, i.e., locations are often subjectively selected based purely on personal judgement.

If any areas show evidence of contamination, such as staining or vegetative stress, <u>biased</u> samples should be collected from each of the areas to characterize the contamination present in each area. If surface drainage patterns such as dry washes or swales are discernable, soil/sediment samples may be collected from the deposits in these features to characterize the immediate areas. Background and control samples are also biased, since they are collected in locations dictated by expected clean conditions or by anticipated impact from adjacent off-site areas.

When soil sampling investigations involve large areas, measured in acres for example, a systematic approach must be taken, not necessarily to the exclusion of other approaches, to characterize the presence and distribution of contaminants. In these situations, a grid-based soil sampling program is employed. There is no single grid size that is appropriate for all sites; however, in most cases, the smaller the site, the smaller the grid size. Common grid sizes are developed on 50-foot and 100-foot centers, although other sizes may be appropriate in given situations. It may be appropriate and acceptable to integrate several different grid sizes in a single investigation.

When the site is extremely large, over several acres for example, it may be impossible to consider sampling every grid and it will be necessary to statistically select a sub-set of the total number of grids in order to reduce the number of samples collected for the study. On the other hand, it may sometimes be appropriate to sample every grid and use relatively inexpensive and quick screening-level analytical techniques to define the areas which must be sampled and analyzed for a higher level of data quality. Because the screening level

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inlet to atmosphere. The evacuated canister should then be filled at the normal sampling rate with the zero gas.

- Initial flowrates will be determined with a mass flow meter. The initial flowrate and initial vacuum (at least 29 inches of Hg) should be recorded on the sample data sheet. Adjust the flowrate so that at the end of the sampling interval the ending pressure of the canister is approximately 0.9 atm.
- Final flowrates should also be determined with a mass flow meter. Final flowrate and final vacuum should be recorded on the sample data sheets. The final vacuum should be between 5 inches and 1 inch of Hg. The final flowrate should be at least 1 scc/min.

After sample collection, all canisters should be double checked to verify that each has an EPA pre-numbered tag with all information filled out. Place the canister in a shipping container and seal the container with EPA sample custody tape.

# 4.13.3 <u>Semi-Volatile Organic Compounds (SVOC) Sampling With High Volume PUF</u> <u>Samplers Using Methods TO-4 & TO-13</u>

4.13.3.1 General -- The following is a synopsis of procedures which should be strictly adhered to for use of the High Volume Polyurethane Foam (PUF) sampling method for sampling semi-volatile organic compounds (SVOC) including pesticides and polychlorinated biphenyls. This summary is adapted from Method TO-4 (pesticides and PCBs) and TO-13 (polynuclear aromatic compounds) of the COMPENDIUM OF METHODS FOR THE DETERMINATION OF TOXIC ORGANIC COMPOUNDS IN AMBIENT AIR.

The following procedures must be followed in preparation of PUF sampling media and using the High Volume PUF method for sampling for SVOCs:

- All PUF sampling media should be pre-cleaned, loaded into High Volume PUF sample cartridges and sealed in solvent washed cans by the extraction lab prior to use.
- Chain-of-custody shall be maintained for all samples.
- 4.13.3.2 <u>PUF Cleaning</u> -- The Air Compliance Unit has responsibility for buying the PUF media and cutting the PUF plugs. PUF media should be specified as <u>not</u> containing any fire retardants. It should be stored in the dark to prevent photo-oxidation. It should be less than two years old, and should be stored in a pesticide free environment.

Care should be exercised in cutting the PUF. It should be thoroughly wet with tap water prior to cutting. A drill press and stainless steel PUF cutting die should be used. The drill press area should be free of oil and a polyethylene cutting block should be used to stop the die at the bottom of the drill press stroke (do not use wood). Water should be sprayed on the die as the PUF is cut to help prevent snagging. After the plugs are cut, they should be

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rinsed with tap water and followed by a rinse with deionized water. Finally, the excess water should be squeezed out.

The rinsed PUF plugs should be placed in a polyethylene plastic bag and delivered to the Analytical Support Branch extraction laboratory for preparation of the PUF/XAD-2 cartridges. The extraction laboratory will be given a minimum of three weeks notice for cleaning and checking the PUF/XAD-2 cartridges. The cleaned PUF/XAD-2 cartridges should be wrapped in aluminum foil and packed in metal cans cushioned by cleaned polyurethane foam to prevent breakage during shipment. Prepared PUF/XAD-2 sample cartridges that are pre-packed in solvent washed metal cans will be obtained from the extraction laboratory prior to sampling. The cans should be packed inside coolers which are lined with polyurethane foam for shipment.

4.13.3.3 <u>Sample Collection</u> -- The following procedures will be followed for all High Volume PUF sampling.

Nylon gloves will be used when handling all PUF cartridges and quartz particulate pre-filters. Assure that the red silicon upper and lower gaskets, located in the cartridge housing, are in place. Then remove the PUF cartridge from the shipping can, remove from the foil and insert the cartridge into the High Volume PUF Sampler's chamber. The pre-filter should be installed in the filter holder using caution not to over tighten the fittings. The foil should be sealed back in the shipping can. The can should be labeled with site ID, operators name, and sample date, and placed in the High Volume PUF sampler enclosure until the sample is collected.

The High Volume PUF sampler should be turned on and allowed to run for two minutes. An initial flowrate should be recorded on the sample data sheet. The timer should be set to turn the sampler on and off at the desired times.

The operator should collect the sample as soon as possible after the sampling period ends. The sampler should be manually turned on and allowed to run for two minutes. A final flowrate should be recorded on the sample data sheet. The final flowrate should be at least 150 liters per minute. The PUF cartridge should be removed, and the quartz pre-filter folded and placed in the top of the PUF cartridge. The PUF cartridge and pre-filter should be re-wrapped in the original aluminum foil and placed back in the shipping can. The can should be tightly sealed. Complete the sample data sheet and Chain-Of-Custody Record and seal the shipping can with a sample custody seal. Finally, the shipping can containing the sample should be placed in a cooler containing frozen eutectic salt packs (at a nominal temperature of  $\sim 4^{\circ}$  C). When all samples are collected from all sites, the cooler should be sealed with sample custody tape for transport back to the ASB.

4.13.3.4 <u>Analytical Support Branch</u> -- Upon arrival of the metal container at the Athens ASB laboratory, the samples shall be stored in the metal container in a refrigerator until submitted to the chemist for extraction.

# 4.13.4 Sampling For Metals Using The High Volume Sampler

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4.13.4.1 General -- The following is a synopsis of procedures which should be strictly adhered to for the sampling of metals in air. This summary is adapted from 40 CFR. PART 50. APPENDIX B - Reference Method For The Determination Of Suspended Particulate Matter In The Atmosphere (High Volume Method). and 40 CFR. PART 50. APPENDIX G - Reference Method For The Determination Of Lead In Suspended Particulate Matter Collected From Ambient Air.

The following procedures must be followed in preparation for collecting samples for metals analyses with the High Volume sampler.

- All filters used will be supplied by the EPA National Filter Distribution Program, and of the same quality as supplied to the State and Local Agency Air Monitoring Stations.
- Prior to use, all filters will be checked for pinholes, and desiccated at 15°C - 30°C, ± 3°C, and less than 50 percent relative humidity, ± 5 percent, for at least 24 hours.
- A filter field blank will be taken to the field, but not exposed. Filter field blanks will be analyzed by the Analytical Support Branch (ASB) to determine the background concentration of metals. The number of filter blanks will be determined based on the number of samples collected, or one blank for each ten samples collected.
- Chain-of-custody must be maintained for all samples.
- 4.13.3.4 <u>Sample Collection Procedures</u> -- Samples will be collected using the High Volume sampler as described, and operated in accordance with 40 CFR, PART 50, APPENDIX B:
  - All flow calibration orifices will be traceable to a Primary Standard Rootsmeter. Flows will be corrected to EPA Standard Temperature and Pressure (25°C and 760 mm Hg).
  - Digital manometers used to determine flow rates will be checked against a U-Tube water manometer prior to use in each study.
  - Air Compliance Unit personnel will remove a 2-inch strip of the exposed filter from one end and discard it. Two 1-inch strips will be cut from the same end and transported to the ASB for analysis.
- 4.13.4.3 <u>Integrated Sample Collection</u> -- The following procedures should be followed to collect time integrated samples:
  - Initial and final flow rates will be determined with a calibrated orifice and a digital manometer.
  - After the sample has been collected, the filter will be folded lengthwise and placed in a filter holder. The filter holder is then placed in an envelope and the envelope sealed.
  - A Chain-Of-Custody Record should be completed which contains the

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time of sampling, the sampling interval, and the signature of the person taking the sample.

After sample collection, all sample envelopes will be placed in an appropriate container. An EPA chain-of-custody seal will be placed on the container. Filters will be cut by the Analytical Support Branch and transported to the ASB for analysis.

# 4.13.5 Sampling and Analysis of Mercury In Ambient Air Using Arizona Instrument Mercury Dosimeter Tubes and the Model 511 Gold Film Mercury Vapor Analyzer

4.13.5.1 General -- The following is a synopsis of procedures which should be strictly adhered to for the sampling and analysis of mercury (Hg) in ambient air. This summary is adapted from the Arizona Instruments® (AZI), Model 511, Gold Film Mercury Vapor Analyzer Manual.

The following generic procedures should be adhered to at all times:

- Chain-of-custody shall be maintained for all samples
- A minimum of one trip blank shall be transported per one to ten samples collected.
- All mercury dosimeter tubes shall be cleaned and analyzed each sample day prior to use.
- 4.13.5.2 <u>Dosimeter Cleanup</u> -- Each dosimeter tube should be connected to the AZI® model 412 dosimeter controller and AZI® Gold Film Mercury Analyzer as shown in Figure 1 of the AZI® model 511 Mercury Analyzer Operation Manual. The mercury analyzer analysis cycle should be started and immediately afterward the dosimeter controller's START button should be depressed. This will cause the gold plated wire inside the dosimeter tube to heat up and liberate any elemental mercury that forms an amalgam on the gold plated wire inside the dosimeter. Continue this cycle until less than 2 ng of Hg is detected from the dosimeter tube. Remove the clean dosimeter tube from the dosimeter controller, tag it with the cleanup date and operators name, and connect the inlet and outlet dosimeter ports together with a clean 8-inch section of 3/16" ID x 1/16" wall Tygon® tubing (this effectively caps the dosimeter tube).
- 4.13.5.3 <u>Sample Collection</u> -- Each sampling pump will be calibrated with a mass flow meter before and after each sampling event. Connected the dosimeter to the battery powered sampling pump with Tygon® tubing. Adjust the sampling pump to obtain a flowrate of 50 cc/min. The sampling interval should not extend beyond 8 hours. The following steps should be followed to collect each sample.
  - Disconnect one end of the Tygon tubing from the dosimeter tube and connect it to the battery powered sampling pump.
  - Program the pump to run the desired number of hours. Position the

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dosimeter tube inlet so that there is no obstruction of flow. Record the dosimeter tube number, site ID, sample date, start time, and initial flowrate on the sample data form.

- As soon as possible after the sampling event, disconnect the Tygone tubing from the sample pump and connect the loose end to the inlet of dosimeter tube. Record the elapsed time from the pump display. Check the ending flowrate with a mass flow meter and record it on the sample data form.
- 4.13.5.4 <u>Sample Analysis</u> -- Before any mercury analysis of dosimeter tubes, the AZI® model 511 should be calibrated. The AZI® model 511 should only be used under laboratory conditions. All glassware should be scrupulously cleaned and used exclusively for mercury analysis. All tygon tubing that is used should be new. The following calibration procedure is adapted from the AZI® model 511 operation manual:

#### Calibration

- Calibration of the AZI® model 511 is accomplished by the reaction of a mercuric chloride standard solution with stannous chloride which liberates elemental mercury. The elemental mercury is sparged from an impinger for analysis in the mercury analyzer.
- Before calibration, a film heat cycle should be initiated to clean the mercury analyzer sensor.
- Fresh working 100 ng/ml mercuric chloride standards should be prepared daily from a 1000 ug/ml stock solution. From the 100 ng/ml working standard, calibration standards of 10 ng/ml, 20 ng/ml, and 40 ng/ml should be prepared. Use the procedure outlined in the AZI® operation manual to introduce the stannous chloride solution into the impinger containing the 40 ng/ml standard. IMMEDIATELY depress the SAMPLE START button on the analyzer. Use the calibration potentiometer to adjust the analyzer to read 40. Analyze the 10, 20, and 40 ng/ml standards to verify that the calibration curve is linear. All analyzer readings should be within 2 ng of the actual values of the standards. Record the readings in the logbook.
- Once the mercury analyzer is calibrated, connect an exposed dosimpter tube to the model 412 dosimeter controller. Connect a zero filter to the inlet of the dosimeter tube. Simultaneously press the mercury analyzer SAMPLE START button and the dosimeter controller START button. Record the analyzer reading (in ng) in the logbook. Repeat this sequence three times and sum the values. This value is the total ng of mercury that was captured on the dosimeter tube.
- Divide the sum of the mercury analyzer readings (in ng) by the total volume of air sampled (in liters) to give the concentration of mercury present in ug/M³.

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# 4.13.6 Dioxin and Dibenzofuran Sampling With High Volume PUF Samplers Using Method TO-9

4.13.6.1 General -- The following is a synopsis of procedures which should be strictly adhered to for use of the High Volume Polyurethane Foam (PUF) sampling method for sampling for polychlorinated dibenzo-p-dioxins and dibenzofurans. This summary is adapted from Method TO-9 of the COMPENDIUM OF METHODS FOR THE DETERMINATION OF TOXIC ORGANIC COMPOUNDS IN AMBIENT AIR.

Since this method requires High-Resolution Mass Spectrometry which ESD does not have, all sample media preparation and analysis will have to be contracted. At least one months notice prior to sampling should be given to the Analytical Support Branch to obtain a contract laboratory program (CLP) contract for any dioxin and dibenzofuran analysis. It is important that the contract specify a number of details to assure accurate results:

- All of the PUF media and a representative number of each batch of quartz pre-filters should be checked by the contract laboratory to assure that there is no contamination. Each PUF plug should be prespiked by the contract laboratory with dioxin and dibenzofuran surrogates as a check of the accuracy of the method.
- Each set of PUF plugs and quartz filters should be securely packed in sealed containers and in coolers to prevent damage during shipment. The sampling media should be shipped air freight to minimize the time between cleanup and sampling.
- Chain-of-custody shall be maintained for all samples.

4.13.6.2 <u>Sample Collection</u> -- The following procedure will be followed for all High Volume PUF sampling for dioxins and dibenzofurans.

Nylon gloves will be used when handling all PUF cartridges and quartz particulate pre-filters. Assure that the red silicon upper and lower gaskets are in place in the PUF cartridge housing. Remove the PUF cartridge from the shipping can. Unwrap and insert the PUF cartridge into the High Volume (Hi-Vol) PUF sampler's chamber. Install the pre-filter in the filter holder using caution not to over tighten the fittings. The removed aluminum foil should be sealed in the shipping container. The container should be labeled with the site ID, the operators name, and the sample date, and placed in the Hi-Vol PUF sampler enclosure until the sample is collected. The Hi-Vol PUF sampler should be turned on and allowed to run for two minutes. An initial flowrate should be recorded on the sample data sheet. The timer should be set to turn the sampler on and off at the desired times.

The operator should collect the sample as soon as possible after the sampling period ends. The sampler should be manually turned on and allowed to run for two minutes and a final flowrate recorded on the sample data sheet. The final flowrate should be at least 150 liters/minute. The PUF cartridge should be removed, and the quartz pre-filter folded and placed in the top of the PUF cartridge. The PUF cartridge and pre-filter should be re-wrapped in the original aluminum foil and placed back in the shipping container and the container should

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be tightly sealed. Complete the sample data and sample custody sheets. Each shipping container should have a sample custody seal. Finally, the shipping container containing the sample should be placed in a cooler containing frozen eutectic salt packs (at a nominal temperature of ~ 4°C). When all samples are collected from all sites, the cooler should be sealed with sample custody tape for shipment to the contract laboratory.

4.13.6.3 <u>Contract Laboratory</u> -- Upon arrival of the metal container at the contract laboratory, the samples shall be stored in the metal container in a refrigerator until submitted to the chemist for extraction.

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# 4.14 CRITERIA POLLUTANT MONITORING (REFERENCE MONITORS)

# 4.14.1 Monitoring Ozone In Ambient Air

4.14.1.1 <u>General</u> -- The following is a synopsis of procedures which should be strictly adhered to for the monitoring of ozone in air. This summary is adapted from 40 CFR 50. <u>APPENDIX D</u> - <u>Measurement Principle and Calibration Procedure For The Measurement Of Ozone In The Atmosphere</u> and the Quality <u>Assurance Handbook for Air Pollution Measurement Systems</u>. Volume II. Section 2.7 (EPA-600/4-77-027a).

The following procedures must be followed in monitoring for Ozone in ambient air:

- Calibration systems will meet 40 CFR, Part 50, Appendix D specifications for a Primary Standard Calibration Photometer.
- Calibration systems will be verified against the National Bureau of Standards (NBS) Standard Reference Photometer #10 before use.
- Monitor enclosures will meet the specifications of monitor reference/equivalent designation for temperature control.
- Probes must meet the requirements stated in 40 CFR, Part 58 for materials and sample residence time.
- All flow calibrations will be traceable to a Primary Standard. Flows will be corrected to EPA Standard Temperature and Pressure (25°C and 760 mm Hg).
- Chain-of-custody must be maintained at all times.
- 4.14.1.2 <u>Monitoring Procedure</u> -- Measurements will be made using the procedure as described, and operated in accordance with 40 CFR, PART 50, APPENDIX D:
  - Monitors will be calibrated at the beginning and end of each study,
     and at least quarterly during the study.
  - Monitors will be calibrated after major maintenance or when a quality assurance (QA) check shows an out-of-control condition exists.
  - A zero/span check will be conducted daily on all monitors.
  - Precision checks of all monitors will be conducted at least weekly.
  - Quality assurance audits as specified in 40 CFR, Part 58, Appendix A will be conducted quarterly, or at least once for short duration studies.

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• Data telemetry systems will be run in parallel with strip chart recorders. The recorder strip chart will serve as a permanent record and diagnostic tool.

After completion of the study, all monitoring equipment will be returned to the Athens, Georgia facility for final check-out. All field documentation will be retained by the Air Compliance Unit.

# 4.14.2 <u>Sampling Of Total Suspended Particulate In Air Using The High Volume</u> Sampler

4.14.2.2 <u>General</u> -- The following is a synopsis of procedures which should be strictly adhered to for the sampling of total suspended particulate in air. This summary is adapted from 40 CFR. PART 50. APPENDIX B - Reference Method For The Determination Of Suspended Particulate Matter In The Atmosphere (High Volume Method). and the Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II. Section 2.2 (EPA-600/4-77-027a).

The following procedures must be followed in sampling for total suspended particulate (TSP) with the High Volume sampler:

- All filters used will be supplied by the EPA National Filter Distribution Program, and of the same quality as supplied to the State and Local Agency Air Monitoring Stations.
- Prior to use, all filters will be checked for pinholes, and desiccated at 15°C - 30°C ± 3°C, and less than 50 percent relative humidity ± 5 percent, for at least 24 hours.
- Initial and final (exposed) filter weights will be determined by the Air Compliance Unit. One in ten filters will be reweighed as a quality assurance check. For batches less than ten, one filter will be reweighed.
- After sampling, filters will be desiccated as described above.
- Chain-of-custody must be maintained for all samples.
- 4.14.2.2 <u>Sample Collection</u> -- Samples will be collected using the High Volume sampler as described, and operated in accordance with, 40 CFR, PART 50, APPENDIX B:
  - All flow calibration orifices will be traceable to a Primary Standard Rootsmeter. Flows will be corrected to EPA Standard Temperature and Pressure (25°C and 760 mm Hg).
  - Digital manometers used to determine flow rate will be checked against a U-Tube water manometer prior to use in each study.
  - Volumetric flow controllers will be used on all high volume particulate samplers. Flows will be determined using the manufacturer's flow rate look-up table supplied with the

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controllers. Flow rate look-up tables will be verified using a calibrated orifice on a routine basis, or at least annually.

- 4.14.2.3 <u>Integrated Sample Collection</u> -- The following procedures should be followed to collect time integrated samples:
  - Initial and final flow rate will be determined with a digital manometer based on the manufacturer's flow rate look-up table.
  - After the sample is collected, the filter will be folded lengthwise and placed in a filter holder. The filter holder is then placed in an envelope and the envelope sealed.
  - A Chain-Of-Custody Record should be completed detailing the time of sampling and the sampling interval, and the signature of the person taking the sample.

After the sample are collected, all sample envelopes will be placed in an appropriate container. An EPA chain-of-custody seal will be placed on the container and the container transported to the Athens, Georgia facility for final weighing of filters.

# 4.14.3 Sampling Of Particulate Matter In Air As PM10

4.14.3.1 General -- The following is a synopsis of procedures which should be strictly adhered to for the sampling of particulate matter as PM₁₀ in air. This summary is adapted from 40 CFR. PART 50. APPENDIX J - Reference Method For The Determination Of Particulate Matter as PM₁₀ In The Atmosphere . and the Ouality Assurance Handbook for Air Pollution Messurement Systems. Volume II. Section 2.11 (EPA-600/4-77-027a).

The following procedures must be followed in sampling for particulate matter as  $PM_{10}$ :

- All filters used will be supplied by the EPA National Filter Distribution Program, and of the same quality as supplied to the State and Local Agency Air Monitoring Stations.
- Prior to use, all filters will be checked for pinholes, and desiccated at 15°C - 30°C ± 3°C, and less than 50 percent relative humidity ± 5 percent, for at least 24 hours.
- Initial and final (exposed) filter weights will be determined by the Air Compliance Unit. One of ten filters will be reweighed as a quality assurance check. For batches less than ten, one filter will be reweighed.
- After sampling, filters will be desiccated as described above.
- Chain-of-custody must be maintained for all samples.

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4.14.3.2 <u>Sample Collection</u> -- Samples will be collected using the High Volume sampler as described, and operated in accordance with, 40 CFR, PART 50, APPENDIX J:

- All flow calibration orifices will be traceable to a Primary Standard Rootsmeter. Flows will be corrected to EPA Standard Temperature and Pressure (25°C and 760 mm Hg).
- Digital manometers used for flow rate determination will be checked against a U-Tube water manometer prior to use in each study.
- Volumetric flow controllers will be used on all PM₁₀ samplers. Flows will be determined using the manufacturer's flow rate look-up table supplied with the controllers (Section No. 4.14.2.2). Flow rate look-up tables will be verified using a calibrated orifice on a routine basis, or at least annually.

4.14.3.3 <u>Integrated Sample Collection</u> -- The following procedures should be followed to collect time integrated samples:

- Initial flow rate will be determined with a digital manometer based on the manufacturer's flow rate look-up table.
- Final flow rate will be determined with the same method.
- After the sample is collected, the filter will be folded lengthwise and placed in a filter holder. The filter holder is then placed in an envelope and the envelope sealed.
- A Chain-Of-Custody Record should be completed detailing the time of sampling and the sampling interval, and the signature of the person taking the sample.

After sample collection all sample envelopes will be placed in an appropriate container. An EPA chain-of-custody seal will be placed on the container and the container transported to the Athens, Georgia facility for final weighing of filters.

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+.15 SATURATION MONITORING (NON-REFERENCE MONITORS)

# 4.15.1 Pm10 Saturation Monitoring Using Non-Reference Portable Monitors

- 4.15.1.1 <u>Introduction</u> -- This document gives procedures for use of the PM₁₀ Saturation Monitors constructed by Lane Regional Air Pollution Control Authority located in Springfield, Oregon. These monitors are not referenced by the U.S. EPA.
- 4.15.1.2 <u>General Description</u> -- Each monitor is shipped with the following items:

Description	Oty
Sampler Body	1
PM ₁₀ Head	1
Filter Holders	2
Rechargeable Battery Packs	2
Battery Charger	1
Sampler Hanger	1
Storage Boxes	2

Procedures for siting requirements, monitor calibration, filter media inspection and preparation, sampler preparation, and battery pack preparation and changing are discussed in Appendix O of the manufacturers Operations Manual.

- 4.15.1.3 <u>Calibrations</u> -- Calibrations will be performed before and after each study for each sampler. Recalibration will be performed before field use on units which require major maintenance. Detailed calibration procedures are given in Appendix 0 of the manufacturers Operation Manual.
- 4.15.1.4 <u>Sampling Procedure</u> -- This sampling technique does not comply with EPA's ambient air monitoring regulations for PM₁₀ sampling. The method has been shown to represent effective and efficient means for the collection of quality PM₁₀ data.

#### Sample Preparation:

- Filters will be kept in protective cassettes. Prior to use, all filters will be visually inspected for defects.
- Each filter will have an ID number assigned to it.
- Each battery will be checked before load-out.

# Sample Collection:

- Record information on Data Sheets.
- Install clean filter, and start system to check for leaks.

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- Set initial flow to 5 L/min, and stop system.
- Input timer program, and verify.
- Input pulse factor into calculator, and start system.

# Sample Retrieval:

- Start system, and record the ending flow rate.
- Note the ending sampling conditions, and record any problems on Data Sheets.
- Remove and replace sample filter following the double bag procedure.

# Sample Preservation:

- Extreme care must be taken in removing exposed filters and reloading of filter heads.
- Filters will be returned to the original sample cassettes, and the ID number verified.
- Complete data sheets and store them and the filter samples in a container sealed with custody tape.
- 4.15.1.5 <u>Sample Custody</u> -- Chain-of-custody will be followed at all times during the sampling study. Chain-of-custody procedures can be found in Section 3.3, of this manual.

# 4.15.2 Carbon Monoxide Saturation Monitoring

RESERVED

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# APPENDIX D STANDARD FIELD ANALYTICAL METHODS

#### METHOD D.1 TEMPERATURE

# D.1.1 Scope and Application

This method is applicable to ground, surface, and saline waters as well as domestic and industrial wastes.

#### D.1.2 Summary of Method

Temperature measurements may be made with any high quality mercury-filled thermometer or thermistor with analog or digital read-out devices.

# D.1.3 Comments

Measurement devices shall be routinely checked against a precision thermometer.

# D.1.4 Test Procedure

- Use only a previously calibrated mercury-filled thermometer or thermistor that has been inspected according to the procedure outlined in Section 6.3.1 - Temperature.
- 2. Allow thermometer or thermistor enough time to equilibrate to outside temperature when removed from a field vehicle.
- 3. Insert thermometer or thermistor in-situ when possible or in a grab sample. Swirl the thermometer or thermistor in the sample and take the temperature reading when the mercury column or read-out needle stops moving; record temperature to the nearest 0.5°C.

#### D.1.5 Precision and Accuracy

Precision and accuracy for this method have not been determined.

# D.1.6 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition p. 126, Method 212 (1985).

Methods for Chemical Analyses of Water and Wastes, US-EPA, 170.1 (1983).

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# METHOD D.2 pH (HYDROGEN ION CONCENTRATION)

# D.2.1 Scope and Application

This method is applicable to ground, surface, and saline waters, as well as domestic and industrial wastes.

# D.2.2 Summary of Method

The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode, and a pH meter.

#### D.2.3 <u>Interferences</u>

- The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.
- Errors due to the presence of sodium at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.
- 3. Coatings of oily material or particulate matter can impair electrode response. Remove these coatings by gentle wiping with a laboratory tissue followed by a distilled water rinse.
- 4. Temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the electrode meter system at the temperature of the samples.
- 5. Poorly buffered solutions with low specific conductance values (less than 200 umhos) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several portions of sample before taking pH measurement.

#### D.2.4 Reagents

Secondary standard buffer solutions (pH 4, pH 7, and pH 10) purchased from commercial vendors shall be used.

# D.2.5 Buffering

- 1. Follow the instructions provided with each type of pH meter.
- 2. Each meter/electrod: system must be buffered at a minimum of two points which bracket the expected pH of the samples. The buffer solutions should be approximately three pH units or more apart. Approximate pH values may be obtained by using multi-range pH paper.

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# D.2.6 Test Procedure

- 1. Allow the meter to equilibrate to ambient temperature when it is removed from a field vehicle.
- Buffer the meter at the temperature of the buffer solution as outlined above in Appendix D.2.5.
- Check sample with pH paper to determine proper pH buffer range.
   Rebuffer meter to proper range if necessary.
- 4. If the sample temperature differs by more than 2°C from the buffer solutions, adjust for the temperature differences.
- Thoroughly rinse the electrode with distilled water.
- 6. Immerse the electrode in-situ when possible or in a grab sample. Swirl the electrode at a constant rate until the meter reading reaches equilibrium. The rate of stirring used should minimize the air transfer rate at the air-water interface of the sample.
- 7. Note and record sample pH; repeat measurement on successive volumes of sample or in-situ until values differ by no more than 0.1 pH unit. Two or three volumes are usually sufficient.
- 8. In the case of low specific conductance samples, such as encountered with some ground waters, add 1 ml of 1M potassium chloride solution per 100 mls of sample and follow steps 6.5 and 6.6.
- 9. When the meter is moved to another sampling location, recheck the meter calibration by inserting the probe into the pH 7 buffer solution and follow operational steps outlined in the owners manual.

#### D.2.7 Apparatus

- Orion Model 399A
- Orion SA 250
- Hydrolab Surveyor II
- YSI 3530, 3500 Water Quality Monitoring System

Note -- Follow the operating instructions for the specific meters listed above.

# D.2.8 Precision and Accuracy

Under normal conditions the accuracy is  $\pm$  0.1 pH unit.

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# D.2.9 References

Standard Methods for the Examination of Wastewater, 16th Edition, p. 429, Method 423 (1985).

Instruction Manual for Models 399 A/F, 399 A/L Analog pH Meter and SA 250, Orion Research Incorporated.

Instruction Manual for Surveyor II, Hydrolab Corporation.

Instruction Manual for YSI Water Quality Monitoring System using the Model 3530 pH electrode assembly.

Annual Book of ASTM Standards, Part 31, "Water", Standard D1293-78(B). Methods for Chemical Analysis of Water and Wastes, US-EPA, 150.1 (1983).

Procedure No. 501, pH Measurement in Low Ionic Strength Solutions, Orion Application Information, Orion Research Incorporated.

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# METHOD D.3 DISSOLVED OXYGEN (MODIFIED WINKLER, FULL BOTTLE TECHNIQUE)

#### D.3.1 Scope and Application

The Winkler dissolved oxygen (DO) method with the azide modification is applicable for use with most wastewater and ground and surface waters that contain nitrate nitrogen and not more than 1 mg/l of ferrous iron. Other reducing or oxidizing materials should be absent.

The azide modification is not applicable for the following samples: (a) samples containing sulfite, thiosulfate, polythionate, appreciable quantities of free chlorine, or hypochlorite; (b) samples high in suspended solids; (c) samples containing organic substances which are readily oxidized by free iodine in an acid solution; (d) untreated domestic sewage; (e) biological flocs; or (f) samples with color which interferes with end point detection. In instances where the azide modification is not applicable, the DO probe should be used.

# D.3.2 Summary of Method

The sample is treated with manganous sulfate, potassium hydroxide, and potassium iodide and finally sulfuric acid. The initial precipitate of manganous hydroxide  $(Mn(OH)_2)$  combines with the DO in the sample to form a brown precipitate, manganic hydroxide  $(Mn(OH)_2)$ . Upon acidification, the manganic hydroxide forms manganic sulfate which acts as an oxidizing agent to release free iodine from the potassium iodide. The iodine, which is stoichiometrically equivalent to the DO in the sample is then titrated with sodium thiosulfate or phenylarsine oxide (PAO).

# D.3.3 Interferences

There are a number of interferences to the DO test which are mentioned above in D.3.1. Most of the common interferences in the Winkler procedure may be overcome by use of the membrane electrode method (D.4).

#### D.3.4 Sample Handling

Where possible, collect the sample in a 300-ml BOD bottle. Special care should be used to avoid entrainment of atmospheric oxygen or loss of DO.

Sample should be collected with a DO Dunker (APHA-type) at depths less than five feet. A Kemmerer type sampler is recommended for depths greater than five feet.

When a Kemmerer sampler is used, the BOD sample bottle should be filled to overflowing by inserting the outlet tube of the sampler to the bottom of the BOD bottle; the tube is slowly withdrawn as the bottle is allowed to overflow. Care must be taken to prevent turbulence and the formation of bubbles when filling the bottle.

If an APHA-type DO Dunker is not available and a shallow depth sample is needed, a bucket may be used to collect a sample of water. A siphon tube should be used to fill the BOD bottle. Coil the tube in the bucket to fill the tube.

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Place one end of the tube near the bottom of a BOD bottle and allow the water to fill and overflow the bottle as outlined in Appendix D.3.4.

#### D.3.5 Reagents

Three bottles are used to contain the reagents. They are labeled (1) manganous sulfate solution, (2) alkaline-iodide-azide solution, and (3) concentrated sulfuric acid. Starch solution and sodium thiosulfate or PAO (0.0375N) are stored in separate bottles.

## D.3.6 Test Procedure

- To the sample in the BOD bottle, add 2 mls of manganous sulfate solution followed by 2 mls of alkaline-iodide-azide solution; stopper with care to exclude air bubbles, and mix well by inverting the bottle several times.
- 2. When the precipitate settles, add 2 mls of concentrated sulfuric acid, re-stopper the bottle and mix by inverting the bottle several times. Complete the analysis within 45 minutes.
- 3. Transfer the entire bottle contents by inversion into a 500-ml wide mouth flask and titrate with 0.0375N thiosulfate solution or PAO to a pale straw color. Add 1-2 ml of starch solution and continue to titrate to the first disappearance of the blue color.

Note: Occasionally, a dark brown or black precipitate persists in the bottle after the addition of the acid. This precipitate will dissolve if the solution is kept for a few minutes longer than usual; or if particularly persistent, add a few more drops of acid.

### D 3.7 Calculation

Each ml of 0.0375N sodium thiosulfate (PAO) titrant used is equivalent to 1 mg/l DO when the entire contents of the 300-ml BOD bottle are titrated.

To express the results as percent saturation at 760 mm atmospheric pressure, the solubility data in Table 421:1 (Whipple and Whipple, p. 413-414, Standard Methods, 16th Edition 1985), may be used. Carefully measure the temperature at the time of sample collection.

# D.3.8 Precision and Accuracy

Reproducibility of this method is approximately 0.2 mg/l of DO at the 7.5 mg/l level due to equipment tolerances and uncompensated displacement errors.

#### D.3.9 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 418, Method. 421 B (1985).

Annual Book of ASTM Standards; Part 31, "Water," Standard D1589-60(A).

Methods for Chemical Analysis of Water and Wastes, US-EPA, 360.2 (1983).

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# METHOD D.4 DISSOLVED OXYGEN (MEMBRANE ELECTRODE)

# D.4.1 Scope and Application

The membrane electrode (ME) probe method for DO measurements is recommended for those samples containing materials which interfere with the modified Winkler procedure as listed in Section D.3.1 of Method D.3.

The ME probe method may be used as a substitute for the modified Winkler procedure provided that the meter itself is standardized against the Winkler method on samples free of interferences.

#### D.4.2 Summary of Method

The most common ME instruments for determination of DO in water are dependent upon electrochemical reactions. Under steady-state conditions, the current or potential can be correlated with DO concentration. Interfacial dynamics at the ME-sample interface are a factor in probe response and a significant degree of interfacial turbulence is necessary. For precision performance, turbulence should be constant.

Refer to the manufacturer's instructions for calibrating and operating each specific DO meter.

# D.4.3 <u>Interferences</u>

- Dissolved organic materials are not known to interfere in the output from DO probes.
- Dissolved inorganic salts are a factor in the performance of DO probes
- Reactive gases which pass through the ME probes may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Hydrogen sulfide will interfere with ME probes if the applied potential is greater than the half-wave potential of the sulfide ion.
- Dissolved oxygen ME probes are temperature sensitive, and temperature compensation is normally provided by the manufacturer.

# D.4.4 Apparatus

- YSI Model 57 DO Meter
- YSI 5700 Series DO Probe
- Hydrolab Surveyor II

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# D.4.5 Sample Handling

Refer to Appendix D.3.4 (Method D.3).

# D.4.6 Calibration

- 1. Fill a clean bucket with uncontaminated or deionized water and place the ME probe into the bucket. Using the siphon method described in Section D.3.4.4 of Method D.3, fill duplicate BOD bottles and determine the DO by the Winkler method.
- 2. Adjust the meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of the water in the bucket, then calibrate the DO indicator dial to read the average DO concentration of the two samples determined by the Winkler test.

#### D.4.7 Test Procedure

- 1. When making measurements be sure that the ME stirring apparatus is working, adjust the temperature compensator, and read the DO dial to the nearest 0.1 mg/l.
- Keep the probe in water when not in use to prevent the membrane from drying out.
- 3. If the sample temperature is 5°C greater than the calibration temperature, the meter should be recalibrated to the temperature of the sample.
- 4. Recalibrate against the Winkler test when the DO readings show a distinct change in DO levels, or when the probe has been in waters high in sulfide.

# D.4.8 Precision and Accuracy

Manufacturer's specification claims 0.1 mg/l repeatability with ± 1 percent accuracy.

# D.4.9 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition p. 395, Method 421 F (1985).

Methods for Chemical Analysis of Water and Wastes, US-EPA, 360.1 (1983).

Instruction Manual YSI Model 57. Dissolved Oxygen Meter, Science Division, Yellow Springs Instrument Company.

Surveyor II Operating Manual, Hydrolab Corporation.

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# METHOD D.5 SPECIFIC CONDUCTANCE

## D.5.1 Scope and Application

This method is applicable to ground, surface, and saline waters, as well as domestic and industrial wastes.

# D.5.2 Summary of Method

- The specific conductance of a sample is measured by use of a selfcontained conductivity meter, Whetstone bridge-type, or equivalent.
- Samples are preferably analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C.

# D.5.3 Test Procedure

- 1. Follow instructions manual for specific field conductivity meter used.
- 2. Check the meter with two standard solutions of approximate specific conductances of 100 and 1,000 umhos/cm, or standards that bracket the expected sample conductance. If the meter does not read within one percent of the standards, determine what the problem is and correct it before proceeding. Most field instruments read conductivity directly; with those instruments, follow the manufacturer's instructions. Report the results to the nearest ten units for readings under 1,000 umhos/cm and the nearest 100 units for readings over 1,000 umhos/cm.
- 3. Record the actual sample temperature when the measurement is made. The meter reading should be converted to specific conductance at 25°C using the information in the manufacturer's instruction manual.

# 5.4 Apparatus Section

- Beckman SoluBridge® Model RB-5/RB-6
- YSI Model 3530 Flow Through Cell
- Hydrolab Corporation Surveyor II

# D.5.5 Precision and Accuracy

The conductivity meters listed above have an accuracy of  $\pm$  2 percent of reading. With satisfactory equipment, results within 1 percent of the true value should be obtained.

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# D.5.6 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 76, Method 205 (1985).

Annual Book of ASTM Standards, Part 31, "Water," Standard D1125-64, P. 120.

Methods for Chemical Analysis of Water and Wastes, US-EPA, 120.1 (1983).

Instruction Manual, SoluBridge® RB-5/RB-6, Beckman Instruments, Inc., Rev. January 1982.

Surveyor II Operating Manual, Hydrolab Corporation, Rev. A February 1985.

YSI Model 3560 Water Quality Monitoring System Instructions, July, 1988.

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# METHOD D.6 CHLORINE, TOTAL RESIDUAL (TITRIMETRIC, AMPEROMETRIC)

### D.6.1 Scope and Application

The amperometric titration method applies to all types of waters and wastes that do not contain a substantial amount of organic matter.

# D.6.2 Summary of Method

Chlorine (hypochlorite ion, hypochlorous acid) and chloramines stoichiometrically liberate iodine from potassium iodide at pH 4 or less.

The iodine is titrated with standard reducing agents such as sodium thiosulfate or PAO using an amperometric meter to determine the end point.

The results are calculated as mg/l Cl even though the actual measurement is of total oxidizing power. This is because chlorine is the dominant oxidizing agent present.

# D.6.3 Interferences

- Vigorous stirring can lower chlorine values by volatilization.
- If necessary, dilute with chlorine demand free water. Water containing organic compounds will reduce the chlorine.
- Copper and silver poison the electrode.

#### D.6.4 Apparatus

- Fisher Porter Model 17T2000 amperometric titrator.
- A l ml microburet.

# D.6.5 Reagents

- Phenylarsine oxide (PAO) (0.00564N), commercially available.
   Standardize with potassium bilodate.
- Five percent potassium iodide (KI) solution (50 gms/l).
- Acetate buffer solution (pH 4).

# D.6.6 Procedure

- Place 200 mls of sample in the sample container.
- 2. Attach to the electrode assembly.
- Add 1.0 ml KI solution.
- 4. Add 1.0 ml acetate buffer.

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- Titrate with 0.00564N PAO.
- 6. As each increment is added, the needle deflects toward the left. When the needle no longer deflects, subtract the last drop added from the buret reading to obtain the mg/l Cl. Less and/or slower deflection signals that the end point is near.

# D.6.7 Calculations

For 0.00564N PAO and a 200-ml sample there are no calculations. The buret reading is in mg/l. The last increment, when the needle does not reflect toward the left, must be subtracted.

# D.6.8 Precision and Accuracy

Precision and accuracy of the procedure is  $\pm 0.03$  mg/l at a concentration of 0.41 mg/l for domestic sewage and 82 percent recovery.

# D.6.9 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 303, Method 408 C (1985).

Annual Book of ASTM Standards, Part 31, "Water," Standard D 1253-76(A).

Methods for Chemical Analysis of Water and Wastes, US-EPA, 330.1 (1983).

Instruction Bulletin for Model 17T2000 Amperometric Titrator, Fisher and Porter Company.

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# METHOD D.7 CHLORINE, TOTAL RESIDUAL (TITRIMETRIC, BACK-IODOMETRIC) (STARCH OR AMPEROMETRIC END POINT)

# D.7.1 Scope and Application

The iodometric back titration method is applicable to all types of waters, but is primarily used for wastewater because it eliminates any contact between the full concentration of liberated iodine and the wastewater.

# D.7.2 Summary of Method

- Chlorine (hypochlorite ion, hypochlorous acid) and chloramines stoichiometrically liberate iodine from potassium iodide at pH 4 or less.
- The iodine quantitatively oxidizes a standardized reducing agent such as sodium thiosulfate or PAO.
- The excess reducing agent is then determined by titrating with a standard iodate (0.00564N) solution. The starch end point color change is from clear to blue.
- A subtraction of the excess amount of reducing agent is included in the calculations and the results are reported as mg/l Cl even though the actual measurement is of total oxidizing power. This is because chlorine is the dominant oxidizing agent present.

#### D.7.3 Interferences

- Manganese, iron, and nitrite interference may be minimized by buffering to pH 4 before the addition of KI.
- High concentrations of organics may cause uncertainty of the end point. This uncertainty can be reduced by acidifying to pH 1.0 if manganese, iron, or nitrite are absent.
- Turbidity and color may make the end point difficult to detect in the back idometric starch method. Practice runs with spiked samples may be necessary.

# D.7.4 Apparatus

- Fisher Porter Model 17T2000 amperometric titrator.
- Standard laboratory glassware.
- A 1.0 ml microburet.

# D.7.5 Reagents

PAO solution (0.00564N); standardize with potassium biiodate.

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- Standard iodate solution (0.00564N).
- Starch indicator.
- Phosphoric acid (10 percent).
- Potassium iodide (KI) five percent solution.

#### D.7.6 Procedure

- Analyze 200 mls of distilled water (blank) prior to the analysis of the sample.
- 2. Starch iodide end point.
  - a. Pipet 5 mls of 0.00564N PAO solution into the flask.
  - b. Add 1 ml five percent KI solution and 2 mls of 10 percent phosphoric acid solution.
  - c. Add 200 mls of sample.
  - d. Mix well.
  - e. Add approximately 1 ml of indicator (starch).
  - f. Titrate with 0.00564N potassium iodate solution to the first appearance of a blue color.

#### 3. Amperometric End Point

- a. Perform steps a through e (above) or follow the directions of the manufacturer of the amperometric titrator.
- b. Place the solution in the proper position on the amperometric titrator.
- c. Titrate with 0.00564N iodate solution: Observe the response of the meter needle. As the end point is approached, the needle will temporarily deflect, then return to or near its original position. Continue dropwise. When the needle deflects and remains deflected, the end point has been exceeded by one drop. As an insurance of the proper end point in clear solutions, add 1 ml of starch solution before beginning the titration. The first appearance of a blue color should correspond to the needle deflection on the amperometric meter. Subtract 1/20th of a ml from the buret reading and record the result.

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# D.7.7 Calculations

$$mg/1 C1 = (A-B) \times 200$$

Where: A - ml of iodate used by the blank

B - ml of iodate used in the titration

C - ml of sample

# D.7.8 Precision and Accuracy

Precision of the method is  $\pm 0.09$  mg/l at a concentration of 1.10 mg/l for domestic sewage and 76 percent recovery.

# D.7.9 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition, p. 300, Method 408 B (1985).

Annual Book of ASTM Standards, Part 31 "Water," Standard D 1253-76(C).

Methods for Chemical Analysis of Water and Wastes, US-EPA, 330.2 (1983).

Instruction Manual for Model 17T2000 Amperometric Titrator, Fisher Porter Company.

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# METHOD D.8 CHLORINE, TOTAL RESIDUAL (DPT COLORIMETRIC - HACH KIT)

#### D.8.1 Scope and Application

N,N-diethyl-p-phenylenediamine (DPD) may be used for natural waters and waters treated with chlorine.

# D.8.2 <u>Summary of Method</u>

- Chlorine (hypochlorite ion, hypochlorous acid and chloramines) stoichiometrically liberate iodine from potassium iodide at pH 4 or less.
- A reaction between the liberated iodine and N,N-diethyl-pphenylenediamine (DPD) produces a red colored solution at a pH of 6.2 - 6.5.
- The solution is spectrophotometrically compared to a series of standards. In the Hach kit colorimeter, the dial face is calibrated to give a direct readout in mg/l chlorine.

# D.8.3 <u>Interferences</u>

- Any oxidizing agents; these are usually present at insignificant concentrations. Oxidized manganese interferes with the DPD reagent (1 ug/1 MnO₄ - 1 ug/1 Cl₂).
- Turbidity and color will essentially prevent the colorimetric analysis.

# D.8.4 Apparatus

- Hach DR-100 colorimeter. The Hach reagents and colorimeter or spectrophotometer are EPA acceptable for NPDES monitoring if used in accordance with approved procedures. The preprinted calibration scales provided by the manufacturer, based upon factors developed under ideal conditions, are only acceptable if verified. The calibration scale must be initially verified using multiple standards and a blank. Each day of use, the calibration scale or curve must be verified with a blank and at least one high and one low standard representative of the linear working range. These standard checks must agree within ± 10% of the original scale or a new curve must be prepared. Verification data should be recorded and maintained on file. See Standard Methods
- 1-cm and 2.5-cm cells.

# D.8.5 Reagents or Standards

 DPD total chlorine powder pillows. The DPD tablets must be discarded if there is evidence of decomposition. The tablets deteriorate rapidly in the presence of moisture, and with age become

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difficult to dissolve. Look for caking and brown color.

NOTE: Do not handle tablets with the hands! The DPD oxalate is toxic; take care to avoid ingestion.

- IN sulfuric acid and IN sodium hydroxide.
- Chlorine demand water. See Standard Methods¹, Method 408 B.3.m for directions for preparing or ASTM, Standard D1193, Consumption of Potassium Permanganate.
- Potassium permanganate stock -- Prepare a stock solution containing 891 mgs/1000 mls.
- Potassium permanganate working stock 10 ppm -- Prepare a working stock solution containing 10 mg/l KMnO, by diluting 10 mls of D.8.5.4 stock solution to 1 liter. Stock is stable for approximately 5 days.
- Potassium permanganate calibration standards -- Prepare calibration standards from the working stock solution and/or KMnO4 calibration standard solutions each day of use.

NOTE: KMnO, standards will fade rapidly, within 15 minutes, when chlorine demand-free water is not used.

# Calibration Standard mg/l mls of Working Stock/100mls

0.05	10.0 of 0.5 mg/l Std.
0.10	10.0 of 1.0 mg/l
0.5	5.0  of  10  mg/1
1.0	10.0 of 10 mg/1
2.0	20.0 of 10 mg/1

#### D.8.6 Procedure Total Chlorine Concentration Range 0-2 ug/l

- 1. Fill a clean 2.5-cm cell to the 10-ml mark with sample.
- Samples should have a pH between 6 and 7. If necessary, adjust pH with 1N sulfuric acid or 1N sodium hydroxide.
- 3. Open a DPD total chlorine powder pillow and add the contents to the sample cell. Cap the cell and swirl to mix. It is not necessary for all the particles to dissolve to obtain an accurate reading. The pH of the sample containing the DPD-buffer pillow must be between 6.2 6.5 units.
- 4. Allow at least three minutes, but not more than six minutes, before moving to the next step.
- 5. Open the light shield, turn the right set knob fully clockwise and place the 1-cm cell holder in the left set position of the sample

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well. Press down firmly to seat the cell holder.

- 6. Hold the button down, meanwhile adjust the left set knob to align the meter needle with the arrow at the extreme left of the scale arc. Remove the cell holder.
- Fill a clean 2.5-cm sample cell with the sample. Cap the cell and place into the cell holder. Press down to seat and close the light shield.
- 8. Set zero on colorimeter by holding the on button down and adjusting the right set knob. Open the light shield and remove the sample cell.
- 9. Fill a clean 1-cm sample cell with the solution from step 2, cap the cell and place it in the cell holder.
- 10. Press the "on" button down and hold until the meter stabilizes. Read and record the mg/l total chlorine from the upper (2.5-cm) scale arc.

# D.8.7 Procedure - Total Chlorine Concentrations of 0-3.5 mg/l

- 1. For total chlorine, 0-3.5 mg/l, follow steps in D.8.6 steps 1-5.
- Follow the directions in D.8.6 step 6 except instead of removing the cell holder, rotate it to the right position.
- Fill a clean 1-cm sample cell with the sample. Cap the cell and place it into the cell holder.
- 4. Set zero as in D.8.6 step 8.
- Fill a clean 1-cm sample cell with the solution from D.8.6 step 2.
- 6. Hold the on button down until the meter stabilizes. Read and record the mg/l total chlorine from the lower (1-cm) scale arc.

# D.8.8 Precision and Accuracy

The precision and accuracy of the method is  $\pm 0.03$  mg/l at a concentration of 1.07 for domestic sewage. The recovery is 100 percent.

#### D.8.9 <u>Calculations</u>

No calculations are required for the kit, the readings are directly in mg/l of total chlorine.

# D.8.10 References

Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, p. 292, Method 408E.

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Methods for Chemical Analysis of Water and Wastes, US-EPA, 330.5 (1979).

Instruction Manual. DR 100 Colorimeter. Model 41100-02. DPD Method for Chlorine, Hach Company, June 1983.

Annual Book of ASTM Standards, Part 31 "Water", ASTM, Standard D 1193, Consumption of Potassium Permanganate.

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#### METHOD D.9 FLUOROMETRIC DETERMINATION OF DYE TRACER

# D.9.1 Scope and Application

This method covers the determination of fluorescence as it relates to commercially available tracer dyes. Rhodamine dyes are fluorescent at a wave length of 590 millimicrons, making them detectable without major interferences in all natural waters.

# D.9.2 Summary of Method

Fluorescent dyes emit light upon irradiation from an external source. The emitted light is proportional to the tracer concentration within the sample.

# D.9.3 Sample Handling

- Since tracers are photoreactive, care should be taken to protect samples from light sources.
- All samples should be stored in glass containers.

# D.9.4 <u>Interferences</u>

- Temperature shifts the fluorescent properties of the tracers; thus all samples should be analyzed at the same temperature as the calibration standards.
- Natural conditions such as the presence of chlorophyll or tannins and lignins in the waters to be traced can impart background fluorescence. Calibration standards should be made from these ambient waters to account for any potential background.
- Sample turbidity may interfere. In highly turbid waters, accuracy may be enhanced by filtration prior to analysis.

#### D.9.5 Apparatus

- Turner Fluorometer Model 10-005.
- Calibration glassware.

# D.9.6 Standards

# D.9.6.1 Flow-Through Configuration

Working stocks (use water sample from study areas as dilution water).

- (A) Dilute 1 ml dye to 1 liter dilution water (solution "A" 1 ppt).
- (B) Dilute 10 mls of solution "A" to 1 liter solution "B" 10 ppm.

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(C) Dilute 100 mls of solution "A" to 1 liter solution "C"- 100 ppm.

#### D.9.6.2 Cuvette or Pour-Through Configuration

Working stocks (use water sample from study area as dilution water).

- (A) Dilute 10 mls dye to 1 liter: rolution "A" 10 ppt
- (B) Dilute 10 mls "A" to 1 liter: solution "B" 100 ppm
- (C) Dilute 1 ml "A" to 1 liter: solution "C" 10 ppm
- (D) Dilute 10 mls to 1 liter: solution "D" 1 ppm

#### From these stocks

- (A) Each ml "B" to 1 liter adds 100 ppb
- (B) Each ml "C" to 1 liter adds 10 ppb
- (C) Each ml "D" to 1 liter adds 1 ppb

#### D.9.7 Procedure

# D.9.7.1 Turner Fluorometer: Model 10-005

- 1. Allow fluorometer to warm up for 10 minutes.
- Using background water, adjust for background fluorescence by setting instrument on most sensitive scale (x31.6 and x100 sensitivity) to read 0.
- 3. Machine circuitry is designed such that one calibration standard, e.g., 100 ppb, produces linear responses throughout a range of 0.05 to 300 ppb.
- 4. Above 300 ppb, emissions from the irradiated dye sample interfere with one another producing a nonlinear condition. Thus, when working above 300 ppb calibration curves are required.
- 5. Even though a single 100 ppb standard produces a linear response in the range of 0.1 to 300 ppb, a second standard, e.g., 10 ppb, should be used as a check.
- 6. Depending upon sensitivity needs, a 100 ppb standard can be used to provide a wide range of tracer concentrations. A typical application by the Branch involves setting a 100 ppb standard to equal 10 on the minimum sensitivity scale (xMS and x100). With this setting, tracer concentrations in the range of 0.05 to 300 ppb can easily be determined.

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# D.9.8 Precision and Accuracy

Precision and accuracy for this method have not been established.

# D.9.9 Reference

Wilson, James F., Jr., <u>Fluorometric Procedures for Dye Tracing: USGS</u>
<u>Techniques for Water-Resources Investigations</u>, Book 3, Chapter Al2 (1968).

Operating and Service Manual. Model 10 Series Fluorometers, Turner Designs, October 1981.

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#### METHOD D.10 SALINITY

#### D.10.1 Scope and Application

This method is applicable for brackish to saline waters having a salinity range of 0 to 40 parts per thousand.

# D.10.2 Summary of Method

The salinity measurement is based upon the direct proportionality between the magnitude of an induced electric current and the electrical conductivity of the water in which it is induced.

#### D.10.3 Comments

Routinely check meter against a resistor which is matched to the meter.

# D.10.4 Test Procedure

- Follow instructions manual for Beckman RS5-3 Portable Salinometer or other salinometer used.
- 2. Record the temperature, specific conductance and salinity as determined. Read salinity to nearest 0.1 ppt.

#### D.10.5 Precision and Accuracy

Beckman Model RS5-3 Portable Salinometer has an accuracy of  $\pm 0.05$  parts per thousand salinity,  $\pm 0.05$ °C temperature, and  $\pm 0.05$  millimhos/cm specific conductance. The Hydrolab Surveyor II Salinometer has an accuracy of  $\pm 0.7$  parts per thousand salinity, 1% full scale for conductivity, and  $\pm$  0.1°C for temperature.

#### D.10.6 Apparatus

Beckman Model RS5-3 Portable Salinometer.

#### D.10.7 References

Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 100 Method 209A (1980).

Instruction Manual, RS5-3 Portable Salinometer, Beckman Instruments, Inc., Revised March 1973.

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# APPENDIX E DESIGN AND INSTALLATION FOR PERMANENT MONITORING WELLS

### E.1 INTRODUCTION

The design and installation of permanent monitoring wells involve the drilling of boreholes into various types of geologic formations that exhibit varying subsurface conditions. Designing and installing permanent monitoring wells in these geologic environments may require several different drilling methods and installation procedures. The selection of drilling methods and installation procedures shall be based on field data collected during a hydrogeologic site investigation and/or a search of existing data. Each permanent monitoring well shall be designed and installed to function properly throughout the entire anticipated life of the monitoring program. When designing monitoring wells the following questions shall be considered:

- What are the short- and long-term objectives?
- · How long will the monitoring program last?
- What contaminants are to be monitored?
- · What types of well construction materials are to be used?
- What kinds of analyses are needed?
- · What are the surface and subsurface geologic conditions?
- What aquifer(s) is going to be monitored?
- Over what depth(s) will the well be screened?
- What is the anticipated total depth of the well?
- What are the general site conditions?
- What are the potential health and safety hazards?
- Are these wells going to serve more than one purpose (i.e., monitoring, pump test, extraction)?

Each of the previous questions can be expanded into many subtopics depending on the complexity of the project. In designing permanent monitoring wells, the most reliable obtainable data shall be utilized. Once the data have been assembled and the well design has been completed, a drilling method(s) has to be selected. The preferred drilling procedure for installing wells is the hollow-stem auger method. However, site conditions may not always be amenable to using the hollow-stem auger method. When this occurs, an alternate method shall be selected that will perform acceptably under the encountered site conditions. It is advisable to select several alternate methods and be prepared to use them if a field problem suddenly occurs that warrants a drilling change.

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The following procedures for designing and installing monitoring wells cover the different aspects of selecting materials, drilling boreholes, and installing monitoring devices. This discussion is presented so that standard practices and procedures will be employed by all EPA staff and contractors who are associated with the design, drilling, and installation of permanent monitoring wells in Region IV.

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# E.2 DRILLING METHODS

The following drilling methods are listed in order of preference; however, final selection shall be based on actual site conditions.

#### E.2.1 Hollow-stem Auger

This type of auger consists of a hollow, steel stem or shaft with a continuous, spiralled steel flight, welded onto the exterior side of the stem, connected to an auger bit, which when rotated, transports cuttings to the surface. This method is best suited in soils that have a tendency to collapse when disturbed.

A monitoring well can be installed inside of hollow-stem augers with little or no concern for the caving potential of the soils and/or water table. However, retracting augers in caving sand conditions while installing monitoring wells can be extremely difficult or impossible since the augers have to be extracted without being rotated. If caving sands are encountered during monitoring well installations, a drilling rig must be used that has enough power to extract the augers from the borehole without rotating them. A bottom plug or pilot bit assembly can be fastened onto the bottom of the augers to keep out most of the soils and/or water that have a tendency to clog the bottom of the augers during drilling. Potable water (analyzed for contaminants of concern) may be poured into the augers (where applicable) to equalize pressure so that the inflow of formation materials and water will be held to a minimum when the bottom plug is released. Rubber "O" rings are normally furnished with new augers to make the augers water "tight"; however, rubber "O" rings are not acceptable for ESD drilling operations. Only Teflon® "O" rings are acceptable. Water-tight center plugs are not acceptable because they create suction when being extracted from the augers. This suction forces or pulls cuttings and formation materials into the augers, thus defeating the purpose of the centerplug. Auguring without a center plug or pilot bit assembly is permitted, provided that the soil plug which is formed in the bottom of the auger is removed when sampling or installing well casings. Removing the soil plug from the augers can be accomplished by washing out the plug using a side discharge rotary bit, or augering out the plug with a solid-stem auger bit sized to fit into the hollow-stem auger. The type of bottom plug or pilot bit assembly proposed for the drilling activity shall be approved by ESD prior to drilling operations. Boreholes can be augered to depths of 150 feet or more (depending on the auger size), but generally boreholes are augured to depths less than 100 feet.

## E.2.2 Solid-stem Auger

This type of auger consists of a solid stem or shaft with a continuous spiralled steel flight, welded onto the stem, and connected to an auger bit. When rotated, cuttings are transported to the surface. This auger method is used in cohesive and semi-cohesive soils that do not have a tendency to collapse when disturbed. Boreholes can be augered to depths of 200 feet or more (depending on the auger size), but generally boreholes are augured to depths less than 150 feet.

Both of the previously discussed auger methods can be used in

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unconsolidated soils and semi-consolidated (weathered rock) soils, but not in competent rock. Each method can be employed without introducing foreign materials into the borehole such as drilling fluids, thus minimizing the potential for cross-contamination. Minimizing the risk of potential cross contamination is one of the most important factors to consider when selecting the drilling method(s) for a project.

#### E.2.3 Rotary Method

This method consists of a drill pipe or drill stem coupled to a drilling bit that rotates and cuts through the soils. The cuttings produced from the rotation of the drilling bit are transported to the surface by drilling fluids which generally consist of water, drilling mud, or air. The water, drilling mud, or air is pumped down through the drill pipe, and out through the bottom of the drilling bit. The cuttings are forced to the surface between the borehole wall and the drill pipe. The drilling fluids not only force the cuttings to the surface but also keeps the drilling bit cool. When considering this method, it is important to evaluate the potential for contamination when fluids and/or air are introduced into the borehole. If the rotary method is selected as one of the drilling methods, water rotary is the preferred method, followed by air rotary and mud rotary.

- E.2.3.1 <u>Water Rotary</u> -- When using water rotary, potable water (that has been analyzed for contaminants of concern) shall be used. If potable water (or a higher quality water) is not available, then potable water will have to be transported to the site or an alternative drilling method must be selected. Water rotary is the preferred rotary method because potable water is the only fluid introduced into the borehole during drilling. Water does not clog the formation materials, thus reducing well development time. The potable water will, however, flow out into the surrounding formation materials (if permeable) and mix with the natural formation water. This mixing of the drilling water and the natural formation water should be evaluated when determining the drilling method. Generally, most of the drilling water will be recovered during well development.
- E.2.3.2 Air Rotary -- When using air rotary, the air compressor shall have an in-line organic filter system to filter the air coming from the compressor. The organic filter system shall be regularly inspected to insure that the system is functioning properly. Air compressors that do not have in-line organic filter systems are not acceptable for air rotary drilling. A cyclone velocity dissipator or similar air containment system shall also be used to funnel the cuttings to one location instead of letting the cuttings blow uncontrolled out of the borehole. The conventional air rotary method does not control cuttings blowing out of the borehole, and is not acceptable unless the above mentioned cyclone velocity dissipator or similar containment system is employed. Any air rotary method that allows cuttings to blow uncontrolled out of the borehole and does not direct them to a discharge point with minimal disturbance shall not be acceptable. Air rotary that employs the dual-tube (reverse circulation) drilling system is acceptable since the cuttings are contained in the drill stems and blown to the surface through the cyclone velocity dissipator and to the ground with little surface disturbance.

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# E.3 BOREHOLE REQUIREMENTS

# E.3.1 Annular Space

The borehole shall be of sufficient diameter so that well construction can proceed without major difficulties. To assure adequate size, a minimum 2-inch annular space is required between the casing and the borehole wall (or the hollow-stem auger wall). For example, an 8-inch borehole is required to install a 4-inch outside diameter (OD) casing. However, if the inside diameter (ID) of the casing is 4 inches, the borehole will have to be larger than 8 inches to include the 2-inch annular space and the outside diameter (OD) of the casing (4-inch ID plus the casing wall thickness). The 2-inch annular space around the casing will allow the filter pack, bentonite pellet seal, and the annular grout to be placed at an acceptable thickness. Also, the 2-inch annular space will allow up to a 1.5-inch diameter tremie tube for placing the filter pack, pellet seal, and grout at the specified intervals. An annular space less than the 2-inch minimum will not be acceptable. When installing a well inside of hollow-stem augers, the inside diameter (ID) of the augers is the area to be considered when determining the 2-inch annular space.

# E.3.2 Overdrilling The Borehole

Sometimes it is necessary to overdrill the borehole so any soils that have not been removed or have fallen into the borehole during auger or drill stem retrieval, will fall to the bottom of the borehole below the depth where the filter pack and well screen are to be placed. Normally, 3 to 5 feet is sufficient for overdrilling. The borehole can also be overdrilled to allow for placement of a sump in the well below the well screen. A sump usually consists of a 5- or 10-foot section of well casing located below the well screen. Sumps serve as catch basins or storage areas for sediment that flows into the well and drops out of suspension and for high density non-aqueous phase liquids. Sumps are added to the well screens when the wells are screened in aquifers that are naturally turbid and will not yield clear formation water (free of visible sediment) even after extensive development. The sediment can then be periodically pumped out of the sump preventing the well screen from clogging or "silting up". If the borehole is overdrilled too much, it can be backfilled to the designed depth with bentonite pellets or the filter sand that is to be used for the filter pack.

# E.3.3 Filter Pack Placement

When placing the filter pack into the borehole, a minimum of 6 inches of the filter pack material shall be placed under the bottom of the well screen to provide a firm footing and an unrestricted flow under the screened area. Also, the filter pack shall extend a minimum of two feet above the top of the well screen. The filter pack shall be placed by the tremie or positive displacement method. Placing the filter pack by "pouring" may be acceptable in certain situations; however, this will be discussed in the next section.

# E.3.4 Filter Pack Seal-Bentonite Pellet Seal (Plug)

A seal shall be placed on top of the filter pack. This seal shall consist

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of a high solids, pure bentonite material. The solids content shall be at least 20 percent. Bentonite materials that have a solids content of 20 percent or greater are available in powder form or in the form of pellets compressed to a density of 70-80 lbs/cu.ft. The preferred method of placing bentonite pellets is by the positive displacement or the tremie method. Use of the tremie method minimizes the risk of pellets bridging in the borehole and assures the placement of pellets (also sand and grout) at the proper intervals. Pouring of the pellets (and filter pack materials) is acceptable in shallow boreholes (less than 50 feet) where the annular space is large enough to prevent bridging and to allow measuring (with a tape measure) to insure that the pellets have been placed at the proper intervals. In order to insure that the pellets have been placed at the proper intervals, the pellets shall be tamped, with an appropriate tamping tool, while the measuring is being conducted. The tamping process minimizes the potential for pellet bridging by forcing any pellets, that have lodged against the borehole wall and/or the well casing, down to the proper interval. bentonite seal shall be placed above the filter pack at a minimum of two feet vertical thickness. The hydration time for the bentonite pellets shall be a minimum eight hours or the manufacturer's recommended hydration time, whichever is greater. In all cases, the proper depths shall be documented by measuring and not by estimating. Other forms of bentonite such as granular bentonite, and bentonite chips have limited applications, and are not recommended for the bentonite seal unless special conditions warrant their use. In any case, deviation from bentonite pellets for the seal, such as a 30 percent solids bentonite grout, shall be approved by a senior staff geologist. If for some reason, the water table is temporarily below the pellet seal interval, potable water (or a higher quality water) shall be used to hydrate the pellets.

# E.3.5 Grouting The Annular Space

The annular space between the casing and the borehole wall shall be filled with either a high solids, pure (no additives), bentonite grout, a neat cement grout, or a cement/bentonite grout. Each type of grout to be used shall be e-aluated as to its intended use and integrity. The grout shall be placed into the borehole, by the tremie method, from the top of the bentonite seal to within 2 feet of the ground surface or below the frostline, whichever is greater. The tremie tube shall have a side discharge port or a bottom discharge port, to minimize damage to the filter pack and/or the bentonite pellet seal, during grout placement. The grout shall be allowed to "set" or cure for a minimum of 24 hours before the concrete surface pad is installed. All grouts shall be prepared in accordance with the manufacturer's specifications. Bentonite grouts shall have a minimum density of 9.4 lbs/gal to ensure proper set-up. The density of the bentonite grouts shall be measured while mixing and no pumping of grout into the borehole will be allowed until the minimum density of 9.4 lbs/gal is attained. In addition, the grouting operation shall not cease until the grout flowing out of the borehole has a minimum density of 9.4 lbs/gal. A mud balance shall be used to measure the specified grout density. Estimating the grout density shall not be acceptable. Drilling muds will not be acceptable for grouting. Cement grouts shall be mixed using 6.5 to 7 gallons of water per 94-lb bag of portland cement (Type I). The addition of bentonite (5-to-10 percent) to the cement grout is for elasticity and the reason for its use shall be documented. The specific mixtures and other types of cements and/or grouts shall be evaluated on a case by case basis.

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### E.3.6 Above Ground Riser Pipe And Outer Protective Casing

The well casing, when installed and grouted, shall extend above the ground surface a minimum of 2.5 feet. In high traffic areas, the well casing may be located below grade, with a water proof cover. A vent hole shall be drilled or cut into the top of the well casing cap to permit pressure equalization, if applicable. An outer protective casing shall be installed into the borehole after the annular grout has "set" for at least 24 hours. The outer protective casing shall be of steel construction with a hinged, locking cap. Generally, an outer protective casing used over a 2-inch well casing is 4 inches square by 5 feet long. Similarly, a protective casing used over 4-inch well casings is 6 inches square and 5 feet long. Round protective casings are also acceptable. A protective casing shall have sufficient clearance around the inner well casing. so that the outer protective casing will not come into contact with the inner well casing after installation. The protective casing shall have a minimum of two These weep holes shall be a minimum 1/4 inch in weep holes for drainage. diameter and drilled into the protective casing just above the top of the level of concrete inside the protective casing to prevent water from standing inside of the protective casing. A protective casing made of aluminum or other soft metals is not acceptable because it is not strong enough to resist tampering. The protective casing is installed by pouring concrete into the borehole on top of the grout. The protective casing is then pushed into the wet concrete and borehole a minimum of 2 feet. Extra concrete may be needed to fill the inside of the protective casing so that the level of the concrete inside of the protective casing is at or above the level of the surface pad. The protective casings shall extend a minimum of 3 feet above the ground surface or to a height so that the cap of the inner well casing is exposed when the protective casing is opened.

#### E.3.7 Concrete Surface Pad

A concrete surface pad shall be installed around each well at the same time as the outer protective casing is being installed. The surface pad shall be formed around the well casing. Concrete shall be placed into the formed pad and into the borehole (on top of the grout) in one operation making a contiguous unit. The protective casing is then installed into the concrete as described in the previous section. The size of the concrete surface pad is dependent on the well casing size. If the well casing is two inches in diameter, the pad shall be 3 feet x 3 feet x 6 inches. If the well casing is 4 inches in diameter, the pad shall be 4 feet x 4 feet x 6 inches. Round concrete surface pads are also acceptable. The finished pad shall be sloped so that drainage will flow away from the protective casing and off of the pad. In addition, a minimum of one inch of the finished pad shall be below grade or ground elevation to prevent washing and undermining by soil erosion. At each site, all locks on the outer protective casings shall be keyed alike.

#### E.3.8 Surface Protection-Bumper Guards

If the monitoring wells are located in a high traffic area, a minimum of three bumper guards consisting of steel pipes 3 to 4 inches in diameter and a minimum 5-foot length shall be installed to a minimum depth of 2 feet below the ground surface in a concrete footing and extend a minimum of 3 feet above ground

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surface. Concrete shall also be placed into the steel pipe to provide additional strength. Steel rails and/or other steel materials can be used in place of steel pipe but approval must be granted by a senior staff geologist or engineer prior to field installation.

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#### E.4 CONSTRUCTION TECHNIQUES

# E.4.1 Well Installation

The borehole shall be bored, drilled, or augered as close to vertical as possible, and checked with a plumb bob or level. Slanted boreholes will not be acceptable unless specified in the design. The depth and volume of the borehole, including the overdrilling if applicable, shall have been calculated and the appropriate materials procured prior to drilling activities. The well casings shall be secured to the well screen by flush-jointed threads and placed into the borehole and plumbed by the use of centralizers and/or a plumb bob and level. Another method of placing the well screen and casings into the borehole and plumbing it at the same time is to suspend the string of well screen and casings in the borehole by means of the wireline on the drill rig. The string of well screen and casings can be placed into the borehole and plumbed in one easy operation. This wireline method is especially useful if the borehole is deep and a long string of well screen and casings nave to be set and plumbed.

No lubricating oils or grease shall be used on casing threads. Teflon tape can be used to wrap the threads to insure a tight fit and minimize leakage. No glue of any type shall be used to secure casing joints. Teflon® "O" rings can also be used to insure a tight fit and minimize leakage; however, "O" rings made of other materials are not acceptable if the well is going to be sampled for organic compounds.

Before the well screen and casings are placed on the bottom of the borehole, at least 6 inches of filter material shall be placed at the bottom of the borehole to serve as a firm footing. The string of well screen and casing shall then be placed into the borehole and plumbed. Centralizers can be used to plumb a well, but centralizers shall be placed so that the placement of the filter pack, bentonite pellet seal, and annular grout will not be hindered. Centralizers placed in the wrong places can cause bridging during material placement. Monitoring wells less than 50 feet deep generally do not need If centralizers are used they should be placed below the well centralizers. screen and above the bentonite pellet seal. The specific placement intervals shall be decided based on site conditions. When installing the well screen and casings through hollow-stem augers, the augers shall be slowly extracted as the sand pack, bentonite seal, and grout are tremied and/or poured into place. The extraction of the augers will allow the materials, being placed through the augers, to flow into the borehole instead of flowing up into the bottom of the augers causing potential bridging problems. After the string of well screen and casing is plumb, the filter material shall then be placed around the well screen (preferably by the tremie method) up to the designated depth. After the filter pack has been installed, the bentonite pellet seal shall be placed (preferably by the tremie method) directly on top of the filter pack up to the designated depth or a minimum of 2 feet above the filter pack. The bentonite pellet seal shall be allowed to hydrate a minimum of eight hours or the manufacturer's recommended hydration time, whichever is longer. After the pellet seal has hydrated for the specified time, the grout shall then be pumped by the tremie method into the annular space around the casings up to within 2 feet of the ground surface or below the frostline, whichever is greater. The grout shall be allowed to set for a minimum of 24 hours before the surface pad and protective

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casing are installed. After the surface pad and protective casing are installed, bumper guards shall be installed (if needed). The bumper guards (a minimum of 3 bumper guards per well) shall be placed around or incorporated into the concrete surface pad in a configuration that provides maximum protection to the well. Each piece of steel pipe or approved material shall be installed into an 8- to 10-inch diameter hole, to a minimum depth of 2 feet below ground surface, and filled with concrete. As previously stated, the bumper guard shall extend above the ground surface a minimum of 3 feet. The total length of each bumper guard shall be a minimum of 5 feet.

After the wells have been installed, the outer protective casing shall be painted with a highly visible enamel paint. Care must be taken not to introduce any paint into the well. The wells shall be permanently marked with the well number, date installed, site name, elevation, etc., either on the cover or an appropriate place that will not be easily damaged and/or vandalized.

If the monitoring wells are installed in a high traffic area such as a parking lot, in a residential yard, or along the side of a road it might be desirable to complete the wells flush with the ground surface and install water-tight traffic covers. Traffic covers are designed to extend from the ground surface down into the concrete plug around the well casing. The covers shall have seals that make the unit water-tight when closed and secured. The traffic covers shall be installed as far above grade as practical to minimize standing water and promote runoff.

# E.4.2 <u>Double Cased Wells</u>

Double cased wells shall be constructed when there is reason to believe that interconnection of two aquifers by well construction may cause cross contamination, and/or when flowing sands make it impossible to install a monitoring well using conventional methods. A pilot borehole shall be bored through the overburden and/or the contaminated zone into the clay confining layer or bedrock. An outer casing ( sometimes called surface or pilot casings) shall then be placed into the borehole and sealed with grout. The borehole and outer casing shall extend into tight clay a minimum of five feet and into competent bedrock a minimum of two feet. The total depths into the clay or bedrock will vary, depending on the plasticity of the clay and the extent of weathering and/or fracturing of the bedrock. The size of the outer casing shall be of sufficient inside diameter (ID) to contain the inner casing, and the 2-inch minimum annular space. In addition, the borehole shall be of sufficient size to contain the outer casing and the 2-inch minimum outer annular space, if applicable. outer casing shall be grouted by either the tremie method or by pressure grouting to within 2 feet of the ground surface. The grout shall be pumped into the annular space between the outer casing and the borehole wall. This can be accomplished by either placing the tremie tube in the annular space and pumping the grout from the bottom of the borehole to the surface, or placing a grout shoe or plug inside the casing at the bottom of the borehole and pumping the grout through the bottom grout plug and up the annular space on the outside of the If the outer casing is set into very tight clay, both of the above methods might have to be used, because the clay usually forms a tight seal in the bottom and around the outside of the casing preventing grout from flowing freely during grout injection. On the other hand, outer casing set into bedrock

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normally will have space enough to allow grout to flow freely during injection. A minimum of 24 hours shall be allowed for the grout plug (seal) to "set" or cure before attempting to drill through it. The grout mixture used to seal the outer annular space can be either a neat cement, cement/bentonite, cement/sand, or a pure bentonite grout. However, the seal or plug at the bottom of the borehole and outer casing shall consist of a Type I portland cement/bentonite or cement/sand mixture. The use of a pure bentonite grout for a bottom plug or seal is not acceptable, because the bentonite grout sets or cures to a gel and is not rigid enough to withstand the stresses of drilling. When drilling through the seal, care shall be taken to avoid cracking, shattering, and/or washing out of the seal, which will be discussed in the next section. If caving conditions exist so that the outer casing cannot be sufficiently sealed by grouting, the outer casing shall be driven into place with a grout seal placed in the bottom of the casing. Removal of outer casings, which are sometimes called temporary surface casings, after well screens and casings have been installed and grouted is not acceptable. Trying to remove outer surface casings after the inner casings have been grouted could only jeopardize the structural integrity of the well.

# E.4.3 Bedrock Wells

The installation of monitoring wells into bedrock can be accomplished in two ways:

1. The first method is to drill or bore a pilot borehole through the soil overburden into the bedrock. An outer casing is then installed into the borehole by setting it into the bedrock, and grouting it into place as described in the previous section. After the grout has set, the borehole can then be advanced through the grout seal into the bedrock. The preferred method of advancing the borehole into the bedrock is rock coring. Rock coring makes a smooth, round hole through the seal and into the bedrock without cracking and/or shattering the seal. Roller cone bits are used in soft bedrock, but extreme caution shall be taken when using a roller cone bit to advance through the grout seal in the bottom of the borehole because "down" pressure can cause cracking, excessive water and eroding(washing), and/or shattering of the seal. Low volume air hammers have been used to advance the borehole, but they have a tendency to shatter the seal because of the hammering action. Any proposed method will be evaluated on its own merits, and will have to be approved by a senior staff geologist before drilling activities begin. When the drilling is complete, the finished well consists of an open borehole from the ground surface to the bottom of the well. There is no inner casing, and the outer surface casing, installed down into bedrock, extends above the ground surface, and also serves as the outer protective casing. If the protective casing becomes cracked or is sheared off at the ground surface, the well is open to direct contamination from the ground surface and will have to be repaired immediately or abandoned. In some instances, the outer surface casing is cut off at the surface or below the surface, depending on the design, and a separate outer protective casing is installed. Another limitation to the open rock

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well is that the entire bedrock interval serves as the monitoring zone. In this situation, it is very difficult or even impossible to monitor a specific zone, because the contaminants being monitored could be diluted to the extent of being nondetectable. The use of open bedrock wells are generally not acceptable in the Superfund and RCRA programs because of the uncontrolled monitoring intervals. However, some site conditions might exist, especially in cavernous limestone areas (Karst topography) or in areas of highly fractured bedrock, where the installation of the filter pack and its structural integrity are questionable. Under these conditions the design of an open bedrock well may be warranted.

2. The second method of installing a monitoring well into bedrock is to install the outer surface casing and drill the borehole (by the approved method) into bedrock, and then install an inner casing and well screen with the filter pack, bentonite seal, and annular grout. The well is completed with a surface protective casing and concrete This well installation method gives the flexibility of isolating the monitoring zone(s) and minimizing inter-aquifer flow. In addition, it gives structural integrity to the well, especially in unstable areas (steeply dipping shales, etc.) where the bedrock has a tendency to shift or move when disturbed. Omitting the filter pack around the well screen is a general practice in some open rock borehole installations, especially in drinking water and irrigation wells. However, without the filter pack to protect the screened interval, sediment particles from the well installation and/or from the monitoring zone could clog the well screen and/or fill the screened portion of the well rendering it inoperable. Also, the filter pack serves as a barrier between the bentonite seal and the screened interval. Rubber inflatable packers have been used to place the bentonite seal when the filter pack is omitted. This method is not acceptable because the packers have to remain in the well permanently and, over a period of time, will decompose and possibly contribute contaminants to the monitoring zone.

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# 1.5 WELL CONSTRUCTION MATERIALS

Well construction materials are chosen based on the goals and objectives of the proposed monitoring program and the geologic conditions at the site(s). In this section, the different types of available materials will be discussed.

#### E.5.1 Well Screen And Casing Materials

When selecting the materials for well construction, the prime concern shall be to select materials that will not contribute foreign constituents, either by leaching or sorption, into the monitoring zone and compromising the integrity of the well and future analytical data. If the monitoring program is designed to analyze for organic compounds, stainless steel materials shall be used (where If the monitoring program calls for the analyses of inorganic compounds only, then PVC materials may be acceptable. Generally, PVC materials are not acceptable for monitoring organic compounds because of their sorption and leaching properties. Another concern is to select materials that will be rugged enough to endure the entire monitoring period. Site conditions will generally dictate the kind of materials that can be used. A preliminary field investigation shall be conducted to determine the geologic conditions, so that the most suitable materials can be selected. The best grade or highest quality material for that particular application should be selected. Each manufacturer can supply the qualitative data for each grade of material that is being considered. All materials selected for monitoring well installation shall be evaluated and approved by a senior staff geologist prior to field activities.

Well screen and casing materials generally used in monitoring well construction on RCRA and Superfund sites are listed in order of preference:

- (1) Stainless Steel (304 or 316)
- (2) Rigid PVC meeting NSF Standard 14 (NSF WC)
- (3) Other (where applicable)

There are other materials used for well screens and casings such as black iron, carbon steel, galvanized steel, and fiberglass, but these materials are not recommended for use in long term monitoring programs on hazardous waste sites because of their low resistance to chemical attack and constituent distribution to the ground water.

In addition to material selection, the minimum diameter for well screens and casings used for permanent monitoring wells shall be 2 inches (inside diameter) (ID). The wall thickness has to be considered when selecting the 2-inch well screen and casing, because a 2-inch ID screen or casing having a total wall thickness greater than 1/8 inch will make the outside diameter (OD) 2 1/4 inches which will reduce the required 2-inch annular space. This is especially true for PVC and Teflon. Schedule 5 stainless steel, which is commonly used for permanent monitoring wells has a very thin wall thickness (approximately 1/16 inch thick) which reduces the 2-inch annular space by only 1/8 inch. However, all minimum requirements for well design and installation shall be adhered to when selecting the appropriate materials. For example, if the ID of the screen at least 6 1/2 inches and the OD is 2 1/2 inches, then the borehole will have to at least 6 1/2 inches in diameter to satisfy the minimum requirements.

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The length of well screens in permanent monitoring wells should be long enough to effectively monitor the interval or zone of interest. However, well screens designed for long-term monitoring purposes shall normally not be less than 5 feet in length. Well screens less that 5 feet long are routinely acceptable in temporary monitoring wells where ground water samples are collected for screening purposes only.

### E.5.2 Filter Pack Materials

The filter pack materials shall consist of clean, well-rounded-to-rounded, hard, insoluble particles of siliceous composition. The required grain-size distribution or particle sizes of the filter pack materials shall be selected based upon a sieve analysis conducted on the soil samples collected from the aquifer materials and/or the formation(s) to be monitored. Filter pack materials shall not be acceptable unless proper documentation can be furnished as to the composition, grain-size distribution, cleaning procedure, and chemical analysis. If a data search reveals that there is enough existing data to adequately design the well screen and filter pack, then it may not be necessary to conduct a sieve analysis on the formation materials to be monitored. However, all data and design proposals will be evaluated and approved by senior staff geologist before field activities begin.

# E.5.3 Filter Pack And Well Screen Design

The majority of monitoring wells are installed in shallow ground water aquifers that consist of silts, clays, and sands in various combinations. These shallow aquifers are not generally characteristic of sand aquifers used for drinking water. Therefore, a more technical approach rather than an estimative approach shall be taken in the design of filter packs and well screens for monitoring wells. The filter pack and well screen design shall be based (as stated above) on the results of a sieve analysis conducted on soil samples collected from the aquifer or the formation(s) that will be monitored. The data from the sieve analysis are plotted on a grain-size distribution graph, and a grain-size distribution curve is generated. From this grain-size distribution curve, the uniformity coefficient (Cu) of the aquifer material is determined. The Cu is the ratio of the 60 percent finer material (D60) to the 10 percent finer material (D10)

The Cu ratio is a way of grading or rating the uniformity of grain size. For example, a Cu of unity means that the individual grain sizes of the material are nearly all the same, while a Cu with a large number means a large range of sizes. As a general rule, a Cu of 2.5 or less shall be used in designing the filter pack and well screen.

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Before designing the filter pack and well screen, the following factors shall be considered:

- Select the well screen slot openings that will retain 90 percent of the filter pack material.
- The filter pack material shall be of the size that minimizes head losses through the pack and also prevents excessive sediment (sand, silt, clay) movement into the well.
- A filter material of varying grain sizes is not acceptable because the smaller particles fill the spaces between the larger particles thereby reducing the void spaces and increasing resistance to flow. Therefore, filter material of the same grain size and well rounded is preferred.
- The filter pack design is based on the gradation of the finest aquifer materials being analyzed.

General Steps To Consider In Designing A Filter Pack:

- Construct a grain-size distribution curve on a grain-size distribution graph from the sieve analysis of the aquifer materials. The filter pack design (as stated above) is based on the gradation of the finest aquifer materials.
- 2. Multiply the D30 size (from the grain-size distribution graph) by a factor of four to nine (Pack-Aquifer ratio). A factor of four is used if the formation is fine-grained and uniform (Cu is less than 3), six if it is coarse-grained and non-uniform; and up to nine if it is highly non-uniform and contains silt. Head losses through filter packs increase as the Pack-Aquifer(P-A) ratios decrease. In order to design a fairly stable filter pack with a minimum head loss, the D30 size shall be multiplied by a factor of four.
- 3. Plot the point from step 2 on the 30 per cent abscissa of a grainsize distribution graph and draw a smooth curve with a uniformity coefficient of approximately 2.5.
- 4. A curve for the permissible limits of the filter pack is drawn plus or minus 8 per cent of the desired curve with the Cu of 2.5.
- 5. Select the slot openings for the well screen that will retain 90 per cent or more of the filter pack material.

The specific steps and procedures for sieve analysis and filter pack design can be found in soil mechanics, ground water, and water well design books. The staff geologists and/or engineers shall be responsible for the correct design of the monitoring wells and shall be able to perform the design procedures.

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#### E.6 SAFETY PROCEDURES FOR DRILLING ACTIVITIES

A site health and safety plan shall be developed and approved by the Health and Safety Officer or designee prior to any drilling activities, and shall be followed during all drilling activities. The driller or designated safety person shall be responsible for the safety of the drilling team performing the drilling activities. All personnel conducting drilling activities shall be qualified in proper drilling and safety procedures. Before any drilling activity is initiated, the area shall be surveyed with the necessary detection equipment to locate, flag or mark, all under ground utilities such as electrical lines, natural gas lines, fuel tanks and lines, water lines, etc. Before operating the drill rig, a pilot hole shall be dug (with hand equipment) to a depth of two to three feet to check for undetected utilities or buried objects. Proceed with caution until a safe depth is reached where utilities normally would not be buried. The following safety requirements shall be adhered to while performing drilling activities:

- All drilling personnel shall wear safety hats, glasses, and steel toed boots. Ear plugs are required and shall be provided by the safety officer or driller.
- Work gloves (cotton, leather, etc.) shall be worn when working around or while handling drilling equipment.
- All personnel directly involved with the drilling rig shall know where the kill switches are located in case of emergencies.
- All personnel shall stay clear of the drill rods or augers while in motion, and shall not grab or attempt to attach a tool to the drill rods or augers until they have completely stopped rotating.
- Do not hold drill rods or any part of the safety hammer assembly while taking standard penetration tests or while the hammer is being operated.
- Do not lean against the drill rig or place hands on or near moving parts at the rear of the rig while it is operating.
- Keep the drilling area clear of any excess debris, tools, or drilling equipment.
- Do not climb on the drilling rig while it is being operated or attempt to repair the rig while it is being operated. The driller shall direct the work on the rig.
- Do not move or pickup any drilling equipment unless directed by the driller and/or the project leader.
- Each drill rig shall have a first-aid kit, and fire extinguisher located on the rig quickly accessible for emergencies.
- Work clothes shall be firm fitting, but comfortable and free of

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straps, loose ends, strings etc., that might catch on some moving part of the drill rig.

- Rings or other jewelry shall not be worn while working around the drill rig.
- The drill rig shall not be operated within a minimum distance of 20 feet of overhead electrical power lines and/or buried utilities that might cause a safety hazard. In addition, the drill rig shall not be operated while there is lightning in the area of the drilling site. If an electrical storm moves in during drilling activities, vacate the area until it is safe to return.

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#### E.7 WELL DEVELOPMENT

A newly completed monitoring well should not be developed for at least 24 hours after the surface pad and outer protective casing are installed. This will allow sufficient time for the well materials to "set" and cure before development procedures are initiated. The main purpose of developing new monitoring wells is to remove the residual materials remaining in the wells after installation has been completed, and to try to re-establish the natural hydraulic flow conditions of the formation, disturbed by well construction, around the immediate vicinity of the well. The new monitoring well shall be developed until the column of water in the well is free of yisible sediment, and the phytemperature, and specific conductivity have stabilized. In most cases the above requirements can be satisfied; however, in some cases the pH, temperature, and specific conductivity stabilizes but the water remains turbid. In this case the well may still contain well construction materials, such as drilling mud in the form of a mud cake and/or formation soils, that have not been washed out of the borehole. Excessive or thick drilling muds can not be flushed out of a borehole with one or two well volumes of purge water. Continuous flushing for several days may be necessary to complete the well development. If the well is pumped to near dryness or dryness, the water table shall be allowed to sufficiently recover before the next development period is initiated. Caution should be taken when using high rate pumps and/or large volume air compressors during well development because excessive high rate pumping and high air pressures can damage or destroy the well screen and filter pack. The onsite geologist shall make the decision as to the development completion of each well. All field decisions shall be documented in the field log book.

The following development procedures are generally used to develop monitoring wells:

- Pumping
- Compressed air (with the appropriate organic filter system)
- Bailing
- Surging
- Backwashing ("rawhiding")
- Jetting

The previous methods can be used, both individually and in combination, in order to achieve the most effective well development. The selected development method(s) shall be approved by a senior staff geologist before any well installation activities are initiated.

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#### E.8 WELL ABANDONMENT

When a decision is made to abandon a monitoring well, the borehole shall be sealed in such a manner that the well can not act as a conduit for migration of contaminants from the ground surface to the water table or between aquifers. To properly abandon a well, the preferred method is to completely remove the well casing and screen from the borehole, clean out the borehole, and backfill with a cement or bentonite grout, neat cement, or concrete. In order to comply with state well abandonment requirements, the appropriate state agency shall be notified (if applicable) of monitoring well abandonment. However, some state requirements are not explicit and are very interpretive, so a technically sound well abandonment method shall be designed based on the site geology, well casing materials, and general condition of the well(s).

#### E.8.1 Abandonment Procedures

The preferred method shall be to completely remove the well casing and screen from the borehole. This may be accomplished by augering with a hollow stem auger over the well casing down to the bottom of the borehole, thereby removing the grout and filter pack materials from the hole. The well casing shall then be removed from the hole with the drill rig. The clean borehole can then be backfilled with the appropriate grout material. The backfill material shall be placed into the borehole from the bottom to the top by pressure grouting with the positive displacement method (tremie method). The top two feet of the borehole shall be poured with concrete to insure a secure surface seal (plug). If the area has heavy traffic use, and/or the well locations need to be permanently marked, then a protective surface pad(s) and/or steel bumper guards shall be installed. The concrete surface plug can also be recessed below ground surface if the potential for construction activities exists. This abandonment method can be accomplished on small diameter (one-inch to four-inch) wells without too much difficulty. With wells having six-inch or larger diameters, the use of hollow stem augers for casing removal is very difficult or almost impossible. Instead of trying to ream the borehole with a hollow stem auger, it is more practical to force a drill stem with a tapered wedge assembly or a solid stem auger into the well casing and extract it out of the borehole. Wells with little or no grouted annular space and/or sound well casings can be removed in this manner. However, old wells with badly corroded casings and/or thickly grouted annular space have a tendency to twist and/or break-off in the borehole. When this occurs, the well will have to be grouted with the remaining casing left in the borehole. The preferred method in this case shall be to pressure grout the borehole by placing the tremie tube to the bottom of the well casing, which will be the well screen or the bottom sump area below the well screen. The pressurized grout will be forced out through the well screen into the filter material and up the inside of the well casing sealing holes and breaks that are present. The tremie tube shall be retracted slowly as the grout fills the casing. The well casing shall be cut off even with the ground surface and filled with concrete to a depth of two feet below the surface. If the casing has been broken off below the surface, the grout shall be tremied to within two feet of the surface and then finished to the ground surface with concrete. The surface pad or specified surface protection shall then be installed.

Well casings consisting of PVC material may be more difficult to remove

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from the borehole than metal casings, because of its brittleness. If the PVC well casing breaks during removal, the borehole shall be cleaned out by using a drag bit or roller cone bit with the wet rotary method to grind the casing into small cuttings that will be flushed out of the borehole by the selected drilling fluid. Another method is to use a solid-stem auger with a carbide auger head to grind the PVC casing into small cuttings that will be brought to the surface on the rotating flights. After the casing materials have been removed from the borehole, the borehole shall be cleaned out and pressure grouted with the approved grouting materials. As previously stated, the borehole shall be finished with a concrete surface plug and adequate surface protection, unless directed otherwise.

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#### E.9 CLEANING AND DECONTAMINATION

All drilling rigs, drilling and sampling equipment, backhoes, and all other associated equipment involved in the drilling and sampling activities shall be cleaned and decontaminated before entering the designated drill site. All equipment should be inspected before entering the site to ensure that there are no fluids leaking and that all gaskets and seals are intact. All drilling and associated equipment entering a site shall be clean of any contaminants that may have been transported from another hazardous waste site, thereby minimizing the potential for cross-contamination. Before site drilling activities are initiated, all drilling equipment shall be thoroughly cleaned and decontaminated at the designated cleaning/decontamination area. The following requirements and procedures are to be strictly adhered to on all drilling activities.

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) shall be steam cleaned and wire brushed before being brought on the site to remove all rust, soil and other material which may have come from other hazardous waste sites. The drill rig and/or other equipment associated with the drilling and sampling activities shall be inspected to insure that all oil, grease, hydraulic fluid, etc., have been removed, and all seals and gaskets are intact and there are no fluid leaks. No oils or grease shall be used to lubricate drill stem threads or any other drilling equipment being used over the borehole or in the borehole without EPA approval. If drill stems have a tendency to tighten during drilling, Teflon® string can be used on the drill stem threads. The drill rig(s) shall be steam cleaned prior to drilling each borehole. In addition, all downhole drilling, sampling, and associated equipment that will come into contact with the downhole equipment and sample medium shall be cleaned and decontaminated by the following procedures.

- 1. Clean with tap water and laboratory grade, phosphate-free detergent, using a brush, if necessary, to remove particulate matter and surface films. Steam cleaning and/or high pressure hot water washing may be necessary to remove matter that is difficult to remove with the brush. Hollow-stem augers, drill rods, shelby tubes, etc., that are hollow or have holes that transmit water or drilling fluids, shall be cleaned on the inside and outside. The steam cleaner and/or high pressure hot water washer shall be capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam (200°F plus).
- Rinse thoroughly with tap water(potable)

NOTE: Tap water (potable) may be applied with a pump sprayer. All other decontamination liquids (D.I. water, organic-free water, and solvents), however, must be applied with non-interfering containers. These containers shall be made of glass, Teflone, or stainless steel. This aspect of the decontamination procedures used by the driller will be inspected by the site geologist and/or other responsible person prior to beginning of operations.

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- 3. Rinse thoroughly with deionized water.
- 4. Rinse twice with solvent (pesticide grade isopropanol).
- Rinse thoroughly with organic-free water and allow to air dry.
   Do not rinse with deionized or distilled water.

NOTE: Organic-free water can be processed on-site by purchasing or leasing a mobile deionization-organic filtration system.

NOTE: In some cases when no organic-free water is available, it is permissible (with approval) to leave off the organic-free water rinse and allow the equipment air dry before use.

- 6. Wrap with aluminum foil, if appropriate, to prevent contamination if equipment is going to be stored or transported. Clean plastic can be used to wrap augers, drill stems, casings, etc., if they have been air dried.
- 7. All downhole augering, drilling and sampling equipment shall be sandblasted before Step #1 if painted, and/or if there is a buildup of rust, hard or caked matter, etc., that can not be removed by steam and/or high pressure cleaning. All sandblasting shall be performed prior to arrival on site.
- 8. All well casing, tremie tubing, etc., that arrive on-site with printing and/or writing on them shall be removed before Step #1. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can supply materials without the printing and/or writing if specified when materials are ordered.
- 9. Well casing, tremie tubing, etc., that are made of plastic (PVC) shall not be solvent rinsed during the cleaning and decontamination process. Used plastic materials that cannot be cleaned are not acceptable and shall be discarded.

Cleaning and decontamination of all equipment shall occur at a designated area on the site, downgradient, and downwind from the clean equipment drying and storage area. The cleaning and decontamination area shall contain a wash water and/or waste pit excavated either with a backhoe or other heavy equipment. The pit and surrounding area shall be lined with heavy duty plastic sheeting and designed to promote runoff of the wash/rinse water into the pit. If a pit cannot be excavated, a catch basin can be constructed out of wood and lined with plastic to contain the waste/rinse water until it can be containerized. All cleaning of drill rods, auger fights, well screen and casing, etc., will be conducted above the plastic sheeting using saw horses or other appropriate means. At the completion of the drilling activities, the pit shall be backfilled with the appropriate material designated by the site project leader, but only after the pit has been sampled, and the waste/rinse water has been pumped into 55-gallon drums for disposal. No solvent rinsates will be placed in the pit unless prior

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approval is granted. All solvent rinsates shall be collected in separate containers for proper disposal.

Tap water (potable) brought on the site for drilling and cleaning purposes shall be contained in a pre-cleaned tank of sufficient size so that drilling activities can proceed without having to stop and haul water. A stainless steel water tank with a minimum capacity of 1,000 gallons is preferred.

All materials used in the drilling activities shall be sampled for QA/QC purposes. These materials include drilling mud (dry and wet), filter pack materials, bentonite pellets, grout (wet and dry), and the tap water from the storage tank. Other QA/QC samples shall be collected such as equipment rinse blanks, field blanks, etc., in accordance with Sections 4 and B.2.

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#### E.10 DRILLING LOG

A system of logging all pertinent data collected during drilling operations shall be maintained. The test hole locations should be recorded and referenced to the site map and/or datum base so that each location can be permanently established. It is imperative that drilling logs be concise, complete, and described in a manner that is easily understood to all who read them. The following items shall be included in the logging data:

- hole number and location;
- description of soils and subsurface conditions (if applicable);
- type of drilling equipment, driller, and drilling company (if applicable);
- method of drilling;
- type and size of casing;
- type and size of well screen;
- depth to will screen;
- type of pump and pumping rate;
- drilling and sampling times;
- depth to water table, and date and time measured;
- type of samples taken and depths from which taken;
- volume of water purged;
- type of well (permanent or temporary);
- type of sampling equipment and/or cleaning procedure; and
- depth of sampling and description (if applicable).

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## APPENDIX F PUMP OPERATING PROCEDURES

In all of the following procedures, it should be understood that all water level and total depth measurements will be determined and purge volumes calculated according to the procedures described in Section 4.9, Ground Water Sampling, and in Section 7.7, Ground Water Level Measurements.

#### F.1 PERISTALTIC PUMP

#### F.1.1 General

When relatively small volumes of water are required for purging and sampling, and the water level is within the limit of suction (generally around 25 feet vertical separation between the pump and water surface) peristaltic pumps can be used. These pumps are generally small, light-weight and portable and are powered by 12-volt batteries. The Branch maintains a supply of charged 12-volt motorcycle batteries to power these pumps.

The application of these pumps differs with respect to purging and sampling. The following sections detail the use of peristaltic pumps for both purposes.

#### F.1.2 Purging With a Peristaltic Pump

- 1. Place a coil of standard cleaned Teflon® tubing, equal to the well depth plus an additional five to ten feet, in a standard cleaned bucket or box which has been lined with clean plastic sheeting or a garbage bag. Enough tubing is needed to run from the ground surface up to the top of the well casing and back down to the bottom of the well. This will allow for operation of the pump at all possible water level conditions in the well.
- Place one end of the tubing into the vacuum side of the peristaltic pump head. Proper sizing of the Teflon® and silastic or Tygon® tubing should allow for a snug fit of the Teflon® tubing inside the flexible tubing mounted in the pump head.
- 3. Run a short section of tubing (does not have to be Teflon®) from the discharge side of the pump head to a graduated bucket.
- 4. Place the free end of the coil of Teflon® tubing into the well until the end of the tubing is several feet below the top of the water column.
- 5. Secure the Teflon® tubing to the well casing or other secure object using electrician's tape or other suitable means. This will prevent the tubing from being lost in the well should all of the tubing be deployed and come loose from the pump head.
- 6. Turn on the pump to produce a vacuum on the well side of the pump

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head and begin the purge. Observe the pump direction to ensure that a vacuum is being applied to the sample/purge line. If the sample/purge line is being pressurized, either switch the tubing at the pump head or reverse the polarity of the cables on the pump or on the battery.

- 7. Purge the well according to the criteria described in Section 4.7 of this manual. If the pumping rate exceeds the recovery rate of the well, continue to lower the tubing into the well several feet at a time, as needed, until the drawdown stabilizes or the well is evacuated to dryness.
- 8. For wells which cannot be evacuated to dryness, particularly those with recovery rates equal to or very nearly equal to the purge rate, there may not be complete exchange and removal of stagnant water in that portion of the water column above the tubing intake. For this reason, it is important that the tubing intake be placed in the very uppermost portion of the water column while purging. Standard field measurements should frequently taken during this process to verify adequacy of the purge (See Section 4.7 for specific details regarding purge adequacy measurements).

#### F.1.3 Sampling With a Peristaltic Pump

The flexible tubing used in peristaltic pump heads does not meet ESD requirements for sampling equipment materials of construction for organic compound sampling and, practically speaking, cannot be field cleaned between sample locations prior to collecting samples for other parameters. For these reasons, it is necessary to use a vacuum container, placed between the pump and the well for sample collection with a peristaltic pump. This method is detailed in the following steps.

NOTE: Samples for purgeable organic analyses cannot be collected using this method. If samples for purgeable organic compound analyses are required, they must be collected with a Teflon® or stainless steel bailer or by other approved methods.

- 1. Disconnect the purge tubing from the pump. Make sure the tubing is securely attached to the protective casing or other secure object.
- 2. Cut off that portion of the tubing that was in contact with the flexible tubing in the pump head.
- 3. Insert the tubing into one of the ferrule nut fittings of a Teflon® vacuum container transfer cap assembly.
- 4. Place a suitable length of Teflon® tubing between the remaining transfer cap assembly ferrule nut fitting and the vacuum side of the flexible tubing in the peristaltic pump head. Securely hand tighten both fittings.

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- 5. Turn on pump. Water should begin to collect in the transfer container (typically a 1-gallon sample container) within a few minutes. If water does not begin to flow into the container within five minutes, check the transfer cap fittings and make sure the assembly is tightly attached to the container. It may be necessary to tighten the ferrule nuts with a wrench or pliers to achieve a vacuum in the system.
- 6. When the transfer container is nearly full, turn off the pump, remove the transfer cap assembly, and pour the sample into the appropriate containers. Samples to be analyzed for extractable organic compounds, metals, and cyanide can be collected using this system. Because the one-gallon (4-liter) containers used by the Branch are rinsed with nitric acid during cleaning, they cannot be used for collecting samples to be analyzed for nitrogen sensitive parameters. Also, as previously noted, samples for purgeable organic analyses cannot be collected in this manner.
- 7. If additional sample volume is needed, replace the transfer cap assembly, turn the pump on, and collect additional volume. The use of Teflon® valves or ball check devices to retain the water column in the sample delivery tubing during the transfer phase, when large volumes of sample are required, is acceptable. These devices, however, must be constructed so that they may be completely disassembled and cleaned according to the procedures in Appendix B.3.
- 8. When sampling is completed, all Teflon® tubing should be discarded and not reused.

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#### F.2 FULTZ® PUMP

#### F.2.1 General

The Fultz® pump is a small 24-volt DC submersible pump suitable for purging most 2-inch and some 4-inch wells and is available in two different diameters, 1.75 inches and 2.5 inches. Operating depths for these pumps range from approximately 135 feet to 150 feet. Maximum pump rates range from approximately 1.5 gallons per minute, at shallower depths, to less than 0.5 gallon per minute at the maximum operating depth. For any given depth, the 2.5-inch pump has a slightly higher pumping rate, compared to the smaller diameter pump. The pump housing for each pump is constructed of 304 stainless steel and houses a high efficiency electric motor and Teflon® gears (rotors). Water is pulled through a fine-mesh stainless steel screen on the pump head by the meshing rotors and is positively displaced through the discharge hose.

As supplied from the manufacturer, power to the pump is supplied by an internal power pack comprised of four 6-volt gel cell batteries. The manufacturer also offers an external power pack, containing the same array of batteries as the internal supply, and a 24-volt DC generator as optional power sources. ESD has found that the pumps operate at higher rates and for longer periods of time when powered either with the generator or with two 12-volt car or motorcycle batteries connected to provide 24 volts.

#### F.2.2 Operation

- 1.2.2.1 Control Panel Switch Functions -- The following is a list of switch functions found on the control panel of the Fultz® pump:
  - ON Supplies power from selected power source to pump motor.
  - OFF Turns pump off.
  - INTERNAL Selects the internal battery array as the power source for the pump. Note: Because the external sources are more reliable and provide longer service, the internal batteries have been removed from all ESD pumps.
  - EXTERNAL Selects an external power source. Source must be plugged into front panel at exterior source plug.
  - FORWARD Selects forward operating mode, used to pump water from the source.
  - REVERSE Selects reverse operating mode, used to empty water from hose through pump head and to flush silt from pump screen, when clogged.

CAUTION: Always turn power off before changing direction of pump to prevent damage to unit or fuse failure.

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F.2.2.2 <u>Purging Procedures</u> -- The following steps detail the operation of the Fultz® pump when used for purging monitoring wells. This pump is not used for sampling, therefore, no sampling procedures are included.

- 1. Select external power source to be used. If generator is used, plug in to external source jack and place generator as far from the well as possible in the downwind direction. If 12-volt batteries are used, connect batteries with provided cables to provide 24 volts. Bridge the positive post of one battery to the negative post of the other. Next, place the red clip from the main supply cable (the long cable which plugs into the face of the control panel of the pump) on the remaining positive post and place the black clip on the remaining negative post.
- Check pump head to make sure pump and electrical connections are secure.
- Lower pump into well, placing pump head no more than one or two feet below the top of the water column.
- 4. Turn pump on and make sure REVERSE/FORWARD switch is in FORWARD position. If the polarity of the power connection is reversed, the amp meter will deflect to the left and the pump will be running opposite of the selected direction. Make the appropriate change.
- 5. During normal operating conditions, the pump should pull no more than 1.5 to 2.0 amps. Newly replaced rotors may temporarily pull slightly more amps. Check amp meter on control panel to make sure pump is operating in this normal range.
- 6. Listen to the pump, as this is an indication of the amount of water over the pump. As the water level is pulled down, the pitch of the sound will increase and become louder. If the water level is pulled down, lower the pump another one or two feet and continue to observe the sound of the pump.
- 7. If the water level is rapidly lowered, caution must be observed as the pump is lowered in the vicinity of the bottom of the well. In this region, be sure to observe the clarity of the water and the amps being registered on the amp meter. If the water becomes extremely turbid and the amps rise out of the acceptable range, these are indications that the pump has been lowered into silt at the bottom of the well. If this occurs, the pump should be momentarily reversed to dislodge the silt from the screen and rotors. If more volume is required to fully evacuate the well under these conditions, a bailer may be a more appropriate choice for the remainder of the purge.
- 8. After completing the required purge, remove the pump from the well and reverse the motor to empty the pump and hose of all contained water. The pump should be switched off as soon as the last water is

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discharged, as running the pump dry may damage the rotors. This water should be collected with the other purge water and handled appropriately. The pump and wetted portion of the hose may now be decontaminated prior to use at the next sample location.

#### F.2.3 Tips and Precautions

The following tips and precautions should be observed for best performance and operating conditions.

- Watch the hose for kinks as the pump is lowered into the well, particularly checking what remains on the hose frame. Kinks will decrease pump performance and will generally manifest themselves as decreased output with higher amp meter readings. Persistent kinked areas can be repaired by several wraps of duct tape to "round" the hose and provide reinforcement. Badly kinked hose should be "redtagged" for replacement.
- 2. Before going to the field, the pumps performance should be checked. At zero head, a properly operating Fultz® pump should pump 1.1 to 1.2 gallons per minute. If much less than 1.1 gallons per minute is pumped, the rotors should be replaced and the pump re-checked. Worn rotors do not merely decrease the pump rate, they also reduce the operating head of the pump.
- 3. Make sure spare fuses are available. The 1.75-inch diameter pump heads require 2.5 amp fuses. The 2.5-inch diameter pump heads require 5 amp fuses.

#### F.2.4 Rotor Replacement

Remove the five screws that hold the pump head on. Carefully rotate the pump cover at the wire, exposing the rotors. With needle-nose pliers, grip each rotor by a tooth and pull it out. Replace with new rotors by pushing them into place with your thumb. Be careful not to shave off the sides of the teeth on the pump body. Replace the pump cover and five screws. Gently snug the screws into place and back them off one turn. Place the pump in a bucket of water and, while running, gradually tighten the screws. This will wear off any burrs on the rotors and give the best performance.

#### F.2.5 Trouble Shooting

No Power to Pump

- 1. Loose connection to power supply
- Make sure clips on batteries are snug
- 2. Water leakage into motor
- 2. Return to factory

Pump Output Reduced

1. Hose kinked

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- 2. Rotors worn
- 3. Intake clogged
- 4. Power supply low
- 5. Silt or sediment in water

#### High Amp Meter Reading 1. Pump out of water

- 2. Silt or sediment in water

- 2. Replace rotors
- 3. Reverse pump direction to clear
- 4. Replace batteries
- 5. If too bad, discontinue pump use
- 1. Lower pump into water column
- 2. Watch amp reading. If it exceeds the recommended operating range, reverse direction of pump to clear intake. If this does not work, discontinue pumping and use bailer

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#### F.3 LARGE DIAMETER ELECTRIC SUBMERSIBLE PUMPS

#### F.3.1 General

Pumps included within this category include any of the typical, large diameter (3-inch to 4-inch) electric submersibles, such as Goulds, Grunfos, or Jacuzzi. These pumps are necessary when large amounts of water must be removed from wells such as deep, 4-inch monitoring wells and drilled or bored potable wells.

These pumps are generally powered by 120-volt generators and require a minimum of two persons for operation. As such, utmost care should be observed to ensure the safe operation of this equipment, particularly from an electrical hazard standpoint. The following sections detail the safety and operation of these pumps.

#### F.3.2 Safety

- Place the generator on dry ground or plastic sheeting as far as practical from the well, in the down-wind direction, and ground it. ESD has several grounding kits consisting of a roll of copper wire and a grounding rod. Wet the ground thoroughly with tap water at the grounding location, if dry, and drive the grounding rod several feet into the ground.
- Inspect the electrical cord for frays, breaks, exposed wiring, etc.
- 3. Check the head space of the well for the presence of an explosive atmosphere with a combustible gas meter.
- 4. With the current tripod and spool set-up, a minimum of two people are required to place, retrieve and operate these pumps safely. If they are used without the aid of the tripod, i.e., all electrical and suspension lines are spooled separately, at least three people are needed to successfully lower and raise the pumps.
- 5. Wear rubber safety boots to insulate against shock hazards.
- 6. If purge water is not collected, direct discharge away from the well and generator, preferably downgradient of area.
- 7. Make sure the generator is set to proper voltage.
- Do not add gas or oil to the generator while it is running.
- 9. Carry the generator, gas, and oil in trailer dedicated to this type of equipment. Do not haul this equipment in the back of any passenger vehicle or with any sampling equipment or containers.

#### F.3.3 Pre-loadout Checkout Procedure

- Check the oil and gasoline in generator, filling up as needed. Take generator outside and start. Place a load on the generator, if possible.
- 2. Inspect the pump and all hose, rope and electrical cord and connections.

#### F.3.4 Operation

Erect tripod over well head and load hose spool. Connect pump to steel winch cable. Using winch crank, lower pump, hose and electrical cord into the well. If no tripod is available, lower the pump into the well by hand. This will require at least three people, one to lower pump with the rope, one to feed the hose and cord into the well, maintaining proper tension, and one to feed rope, hose and electrical cord from cart.

NOTE: Keep all hose, electrical cord and cable off of the ground at all times. Do not allow the rope, cord, or the hose to scrape or rub on the well casing.

- Place pump five feet below the top of the water column.
- Start generator, then connect power cord from pump.
- 4. After starting pump, closely observe operation to determine if draw-down is occurring in well. If the water level is not pulled down significantly, keep pump at initial level and continue to purge. If the water level drops, lower the pump to keep up with the drawdown. Do not allow the pump to run dry, as this will damage it.

#### F.3.5 Maintenance and Precautions

- Do not put up wet.
- 2. Empty hose of contaminated water before leaving sample location. Do not bring back hose with water in it.
- Do not pump dry.
- 4. Do not run generator without checking oil.
- Do not put pump in trailer with generator.

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#### F.3.6 Trouble Shooting

No Power to Pump	1. Loose connections at pump	<ol> <li>Check wiring at pump.</li> <li>Repair as needed.</li> <li>(Generator off!!)</li> </ol>
	<ol><li>Cord unplugged at generator</li></ol>	2. Plug pump back in
Generator Running, No Pump Output	1. Pump out of water	1. Lower pump into water
No ramp output	<ol><li>Hose collapsed or kinked</li></ol>	2. Unkink hose
	3. Generator output failing	<ol> <li>Put load on generator check output or check voltage output meter</li> </ol>
Sluggish Discharge	<ol> <li>Sediment or other material clogging screen</li> </ol>	1. Remove material from screen
	2. Kinked hose	2. Unkink hose

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#### F.4 QED® BLADDER AND PURGE PUMPS

#### F.4.1 General

The Branch has several QED® bladder pumps and purge pumps (no bladder) which can be used for purging monitoring wells. The bladder pumps, when used near the top of the water column, have a very low efficiency and will generally not purge more than 0.5 gallons per minute. The purge pump, however, can achieve pump rates of several gallons per minute in these situations. The efficiency of the bladder pumps is restricted by the rigid Teflon® bladder, which requires significant hydrostatic head for rapid and complete filling. The purge pump, having no bladder, fills much faster under the same conditions.

Both pumps operate by cycling a pressurized gas on and off in a discharge and refill cycle. The gas, usually atmospheric air, is pressurized and regulated by a compressor/regulator combination (controller), which can consist of either a small, battery powered unit, capable of providing pressure to operate the bladder pump at a depth of approximately 75 feet, or a larger, gasoline powered unit that will allow operation at depths of over 150 feet.

#### F.4.2 Operation - Bladder Pump

- Connect air supply hose to "pump supply" connection on controller and to brass air connection on hose reel cart.
- Lower pump into well and place top of pump several feet below the top of the water column.
- Turn on the compressor. If the gasoline powered compressor is used, place as far from the well as possible, in the down wind direction.
- Adjust the timing of the discharge and refill cycles until maximum flow is achieved.
- Lower pump, as necessary, if water level is reduced in well.

#### F.4.3 Operation - Purge Pump

- 1. Connect air supply hose to "pump supply" connection on controller and connection on top of pump. Observe flow direction arrow on purge pump exhaust adapter. The arrow must point in the direction of air flow from the controller to the pump.
- Attach adequate length of standard garden hose to hose fitting at top of pump.
- 3. Lower pump, air hose and garden hose into water column until top of pump is several feet below top of water column.
- 4. Follow Steps 3 through 5 above.

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#### F.4.4 Trouble Shooting

Compressor running, no 1. Air supply fittings pressure on discharge cycle

- loose
- 1. Check all fittings and tighten
- 2. Bladder is perforated
- 2. Replace bladder
- 3. Exhaust adapter installed 3. Remove adapter and in wrong direction
- replace in correct orientation

Compressor running, pressure low, no water discharged

- 1. Obstruction in ball check assembly allowing water to be pushed out of pump at check
- 1. Remove obstruction
- 2. Air supply fittings loose 2. Check all fittings

#### APPENDIX G AIR MONITORING SAFETY EQUIPMENT CALIBRATION PROCEDURES

#### G.1 GENERAL

#### G.1.1 Introduction

This appendix gives specific procedures to be followed when calibrating air monitoring instrumentation. The calibrations defined in these procedures will result in instrument response accuracy within the capabilities of the instruments. While it is not imperative that the instruments be capable of operating at a high degree of analytical precision and accuracy, it is necessary that calibrations demonstrate proper operation of the monitor and insure that results give an acceptably accurate indication of conditions upon which to base safety decisions and actions.

#### G.1.2 Calibration Gases

All calibration games will be certified by their supplier to be of a specified and known concentration. The concentrations of calibration games will be within a relevant range of response for the air monitors, but will not exceed any flammability or toxic exposure limits. Calibration mixtures and approximate concentrations for specific air monitors will be as follows:

Monitor	Gas Mixture	Concentration
Combustible Gas	Pentane in Air	0.75
Flame Ionization Detector	Methane in Air	75 ppm
Photo-Ionization Detector	Toluene in Air	100 ppm

Gas cylinders will not be sent to the field if they contain less than one-fifth of their full capacity. Cylinders below the required volume will be utilized in the warehouse for equipment checkout and maintenance.

#### G.1.3 Calibration Equipment

All calibrations will consist of introducing a gas of known concentration to the monitor at atmospheric pressure. Under no circumstances will it be acceptable to attempt calibration when the monitor is measuring gas concentrations below or above atmospheric pressure.

To insure stable pressure of the calibration gas, a calibration manifold system will be used. The manifold will consist of a "T" fitting, a Teflon® bag, Teflon® tubing, and fittings. The Teflon® bag is omitted for calibration of the "A. The calibration gas cylinder will be connected to the "T" fitting with lon® tubing so that gas will flow straight through the top of the "T" to a

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Teflon® bag. The "T" fitting and tubing will be purged with calibration gas prior to connection of the Teflon® bag. The bottom or side port of the "T" will be connected via Teflon® tubing to a stainless steel quick disconnect. Once the Teflon® bag has been filled with gas, the gas cylinder flow will be turned off. The monitor's probe will be connected to the manifold via the quick disconnect and allowed to sample the contents of the teflon bag.

#### G.1.4 Calibration Frequency

It is required that monitors be calibrated each time they are turned on. More frequent calibrations are encouraged if samplers feel that field conditions and hazards warrant. Frequent checking of monitor response or proper setting and operation of alarms is encouraged. Prior to turning off the monitor, a post calibration check will be performed. This check will follow the same procedures as the initial calibration except that no adjustments will be made to the monitor. Instead, the response will simple be logged in the field book.

#### G.1.5 Documentation

Calibrations will be documented in the field log book. The entry needs to include the following information:

Date
Time
Time
Monitor's ID #
Battery Check Response
Alarm Response
Fuel Level (FID)
Calibration Gas Concentration
Instrument Response
Operator's Initials

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#### G.2 MSA MODEL 260 COMBUSTIBLE GAS AND OXYGEN ALARM

#### G.2.1 <u>Introduction</u>

The MSA Model 260 Combustible Gas and Oxygen Alarm is a hand-carried, battery operated instrument. It is used to sample atmospheres for combustible gases or vapors and oxygen content and warn the user when pre-determined concentrations of either are reached. The monitor will only detect combustible gases and vapors in air. It will not indicate the presence of combustible airborne mists or dusts such as lubricating oils, coal dust, or grain dust.

## THE LACK OF A RESPONSE ON THIS METER DOES NOT GUARANTEE THAT THE ENVIRONMENT IS SAFE.

#### G.2.2 Operational Checks

- 1. Connect probe line to monitor's water drop-out bottle. Check probe fitting and water drop-out bottle fitting for tightness.
- Place the monitor's "ON-OFF" switch in the "HORN OFF" position. Note that the monitor's "% OXYGEN" and "% LEL" meters respond upscale, then stabilize. Note also, that the oxygen alarm and LEL alarm lights are illuminated, the green flow indicator is flashing, and the "FLOW" indicator float is vibrating audibly.
- Press the "RESET" button and observe that both "ALARM" lights go out.
- 4. Press the "CHECK" button and record the battery reading from the "% LEL" meter.
- 5. Set the "% OXYGEN" meter to read 20.8% using the "CALIBRATE 02" knob. Likewise, set the "% LEL" meter to read zero using the "ZERO LEL" knob.
- Place the monitor's function switch to the "ON" position.
- 7. Leak check the monitor by placing your thumb tightly over the probe line inlet. Observe that the monitor's pump stops. Observe also that when the "% OXYGEN" meter falls to around 19%, the "ALARM" light illuminates and the alarm horn sounds.
- 8. Remove thumb from probe inlet line. When "% OXYGEN" meter returns to 20.8%, press "RESET" button.
- 9. Rotate the "ZERO LEL" knob clockwise until the "ALARM" light illuminates and the alarm horn sounds. This should occur at about 25% of LEL. Return the LEL meter to a reading of zero and reset the alarms.

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#### G.2.3 Calibration

- 1. Assemble a calibration manifold as described in G.1.3. Upon introduction of the calibration gas to the monitor, the LEL response should be around 50%. Record the response.
- 2. Disconnect the monitor from the calibration manifold and reset the alarms.
- Insure that the function switch is in the "ON" position and that the green flow indicator is steadily illuminated.
- 4. Attach probe to the probe line.

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#### .3 CENTURY MODEL OVA-128 ORGANIC VAPOR ANALYZER

#### G.3.1 Introduction

The Century Model OVA-128 Organic Vapor Analyzer is designed to detect organic materials in air. It uses a hydrogen flame ionization detector (FID) as its detection principle. This detector allows the monitor to respond to a wide variety of organic compounds, but limits its sensitivity to around 10 ppm under ideal circumstances.

THE LACK OF A RESPONSE ON THIS METER DOES NOT GUARANTEE THAT THE ENVIRONMENT IS SAFE.

#### G.3.2 Operational Checks

- Connect the hand readout unit's electrical and pneumatic fittings to the side pack assembly.
- Connect probe to the hand readout unit.
- 3. Place the "PUMP" switch in the ON position. Check the battery's condition by placing the "INSTR" switch to the BATT position and observe the response on the hand readout unit.
- Place the "INSTR" switch in the ON position.
- 5. Set the "Calibration Switch" the the "X10" position.
- 6. Use the "CALIBRATE" knob to set the readout to a reading of 6. Using the Alarm Level Adjustment Knob on the back of the readout, obtain an audible response to the reading of 6.
- Set the "Calibration Switch" to the "X1" position.
- 8. Use the "CALIBRATE" knob to set the readout to a reading of 0, and check that the flame-out alarm is audible.
- Place the "PUMP" switch in the ON position and observe that the "SAMPLE FLOW RATE" indicator shows flow.
- 10. Open the "H2 TANK VALVE" and the "H2 SUPPLY VALVE" one turn each. Allow fuel to flow for about 1 minute.
- Press ignitor button and hold until readout unit indicates ignition.
- 12. Use "CALIBRATE" knob to set readout to a reading of 0. (Note: a small positive offset above 0 may be necessary to prevent activation of the flame-out alarm.)

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#### G.3.3 Calibration

- 1. Assemble a calibration manifold as described in G.1.3. using methane as the calibration gas. (Remember to omit the use of a Teflon® bag.)
- 2. Set the "CALIBRATION SWITCH" to the appropriate position for the concentration of the calibration gap. (Usually X10)
- Connect the instrument's probe to the calibration manifold and allow it to sample the calibration gas.
- 4. The readout should indicate close to the concentration of the calibration gas plus any offset which may have been added.
- 5. Place the "CALIBRATION SWITCH" in the "X1" position before entering the site.

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#### G.4 PHOTOVAC TIP II PHOTOIONIZATION DETECTOR

#### G.4.1 Introduction

The Photovac TIP II is designed to detect primarily organic materials in air. It uses a photoionization detector (PID) as its method of operation. The instrument is capable of measuring concentrations down to about 1 ppm sensitivity for certain compounds. It is important to realize that this sensitivity is not achievable for all compounds. Some materials will result in a very low response on the PID in relation to their actual concentrations, while others well not respond at all to the detector's ionization energy. As a general rule, the PID should not be used to monitor for compounds whose structures contain only single bonds.

THE LACK OF A RESPONSE ON THIS METER DOES NOT GUARANTEE THAT THE ENVIRONMENT IS SAFE.

#### G.4.2 Operational Checks

- 1. Press the "POWER" switch to turn the instrument on. After a few seconds, the pump motor should start running.
- Observe that the "LOBAT" (Low Battery) indication is not displayed on the LCD.

#### G.4.3 Calibration

- 1. Unlock the "ZERO" control by turning the locking ring clockwise.
- In a "Background" type of atmosphere, adjust the "ZERO" potentiometer until the LCD reads approximately zero. Return the locking ring to the locked position.
- 3. Assemble a calibration manifold as described in G.1.3 using toluene as the calibration gas.
- Connect the instrument's probe to the calibration manifold and allow it to sample the calibration gas.
- 5. The LCD should indicate close to the concentration of the calibration gas. If not, unlock the "SPAN" control by turning the locking ring clockwise. Adjust the "SPAN" control until the LCD reads approximately the concentration of the calibration gas. Return the locking ring to the locked position.

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#### G.5 HNU MODEL PI 101 PHOTOIONIZATION DETECTOR

#### G.5.1 Introduction

The HNU model PI 101 is designed to detect primarily organic materials in air. It uses a photoionization detector (PID) as its method of operation. The instrument is capable of measuring concentrations down to about 1 ppm sensitivity for certain compounds. It is important to realize that this sensitivity is not achievable for all compounds. Some materials will result in a very low response on the PID in relation to their actual concentrations, while others will not respond at all to the detector's ionization energy. As a general rule, the PID should not be used to monitor for compounds whose structures contain only single bonds.

THE LACK OF A RESPONSE ON THIS METER DOES NOT GUARANTEE THAT THE ENVIRONMENT IS SAFE.

#### G.5.2 Operational Checks

- 1. Connect the probe to the meter case of the instrument.
- Place the function/range switch in the "BATT" position and note the meter's response.
- 3. Place the function/range switch in any of the three range positions. Listen closely to the probe for a humming sound which indicates that the sample fan is operating.

#### G.5.3 Calibra Da

- Place the function/range switch in the "STANDBY" position. Use the "ZERO" potentiometer to adjust the meter reading to zero.
- 2. Assemble a calibration train as described in 6.1.3 using toluene as the calibration gas.
- Place the instrument's function/range switch in the appropriate range for the calibration gas (usually 0-200).
- Connect the instrument's probe to the calibration manifold and allow it to sample the calibration gas.
- 5. The readout should indicate close to the concentration of the calibration gas. If not, use the "SPAN" potentiometer to adjust the meter to the appropriate response.
- 6. Place the function/range switch in the "0-20 ppm" position before entering the site.

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#### G.6 LUDLUM MODEL 3 RADIATION SURVEY METER

#### G.6.1 Introduction

The Ludlum Model 3 is a portable radiation survey meter. The instrument is set for 900 volt Geiger-Mueller Tube (GMT) measurement of beta and gamma radiation. The range of the meter is from 0 to 200 milliroentgen per hour. It is important that the operator realizes that this meter will not respond to most alpha radiation.

THE LACK OF A RESPONSE ON THIS METER DOES NOT GUARANTEE THAT THE ENVIRONMENT IS SAFE.

#### G.6.2 Operational Checks

- Place the multifunction switch in the "BAT" position and note the meter's reading.
- 2. Place the multifunction switch in the "XO.1" position, the F/S switch in the "S" position, and the "AUDIO" switch in the "ON" position. Note that an audible clicking sound can be heard while the meter is counting. After a few seconds, press the "RES" button and note that the meter returns to zero.

#### G.6.3 Calibration

- 1. Read and record the background radiation level.
- Place the GMT probe flat against the casing of a certified Sr90 standard.
- 3. Adjust the multifunction switch until the meter reading remains on scale.
- 4. Read and record the meter's response.
- 5. Calculate the detector's efficiency as follows:

#### E = Meter Reading - Background Activity

- 6. Check that the calculated efficiency is within  $\pm$  0.1 of the efficiency rating placarded on the meter.
- 7. Set the multifunction switch to "XO.1" before entering the site.

## APPENDIX H STANDARD WASTEWATER FIELD METHODOLOGY

#### H.1 GENERAL

Field Methodology at Wastewater Treatment Plants (WWTPs) ranges from actual in-situ measurements of pH and dissolved oxygen (DO), to process control tests such as sludge settleability, sludge blanket depth measurements, and mixed liquor microscopic examinations. Field analytical procedures such as DO, pH, and TRC are covered in detail in Section 6 and Appendix D. Standard sampling procedures, including the collection of samples for trace organic compound analysis, are presented in Section 4 of the SOPQAM.

#### H.2 PROCESS CONTROL TESTING

The "EPA Activated Sludge Process Control Testing" handbook is the standard reference for activated sludge process control testing by Branch personnel (1). Additional references are available that provide a more comprehensive evaluation of the methods used to complete a diagnostic evaluation (DE) at a WWTP (2,3). Completion of the Sacramento "Operation of Wastewater Treatment Plants" course is a requirement for all Branch personnel prior to serving as the project leader on a DE (4). A project leader should also be familiar with the procedures for conducting a comprehensive performance evaluation (CPE) at WWTP (5).

#### H.3 PROCESS CONTROL TESTS

The six basic activated sludge process control tests are:

- Sludge Settleability (settlometer)
- Centrifuge Spins
- Aeration Basin DO Profiles
- Oxygen Uptake Rate (OUR) Measurements
- Mixed Liquor Microscopic Examinations
- Sludge Blanket Depth (SBD) Measurements

For each of the six basic tests the "EPA Activated Sludge Process Control Testing" handbook discusses the objective of the test, equipment needed, procedure, and interpretation of the test results.

#### H.4 REFERENCES

- US-EPA, "Activated Sludge Process Control Testing", ESD, Water Compliance Unit, Athens, GA, 1990.
- US-EPA, "Process Control Manual: Aerobic Biological Treatment Facilities MD-14", EPA 430/09-77-006, Office of Water, Washington, D.C., 1977.

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- 3. Metcalf and Eddy, Inc., "Wastewater Engineering: Treatment, Disposal, Reuse", McGraw-Hill Book Co., New York, NY, 1979.
- 4. California State University Sacramento, "Operation of Wastewater Treatment Plants Volumes I, II, III", Sacramento, California.
- 5. US-EPA, "Retrofitting POTWs", EPA 625/6-89/020, Center for Environmental Research Information, Cincinnati, Ohio, 1989.

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#### APPENDIX I SATURATION MONITORING

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#### I.1 GENERAL DESCRIPTION

Each monitor is received and shipped with the following items:

Description	
Sampler Body	1
PM ₁₀	
Head	1
Filter Holders	2
Rechargeable Battery Packs	2
Battery Charger	1
Sampler Hanger	1
Storage Boxes	2

A general description of the individual components is listed below:

#### I.1.1 Sampler Body

The sampler body is constructed of 6-inch PVC sewer pipe, approximately 18 inches tall. Each end has a PVC cap. The bottom end cap is screwed to the sampler body, and has three triangularly spaced metal connectors. These connectors fit into the battery pack. The bottom cap also has two clasps to hold the battery pack onto the sampler. The top cap serves as the mounting for the circuit board. Access to the board is gained by first removing the bail handle assembly running diametrically through the sampler body. The sampler pump is mounted directly on the circuit board. Also mounted to the top cap is a female quick disconnect fitting for connecting the entire filter assembly.

I.1.1.1 <u>Circuit Board</u> -- Controls for the saturation monitor are located on the circuit board (Figure I.1.1). The four control buttons located near the top are used to start and stop the unit, and to set and program the timer. The calculator is used as a flow totalizer. The four potentiometers (pots) located around the board are used to set different parameters. The rotameter gives an immediate reading of the current flow. Two switches located near the bottom of the board controls battery power and a pulsing circuit. The AA battery is used to power the timer and calculator.

I.1.1.2 <u>Control Buttons</u> -- The four control buttons perform the following functions:

Button	Function
1	By-passes the low battery voltage shutoff
2	Manual ON/OFF control

3 Set4 Function

2 & 4 Pressed together will reset timer & program

## I.1.1.3 <u>Potentiometers</u> (POT) -- The four pots have the following functions:

<u>Pot</u>	Function
1	Controls low battery voltage setting
2	Controls pulse circuit pump off time interval
3	Controls pulse circuit pump on time
	interval
4	Controls pump speed - flow

#### I.1.1.4 <u>Dip Switches</u> -- These two switches:

- Pump power ON/OFF, switch 1
- Pulse Circuit ON/OFF, switch 2
- I.1.1.5 <u>Calculator</u> -- The electronics of the sampler was designed so that by imputing the correct pump calibration figure, the calculator will keep a running total of the units of air flow sampled. Each time the pump rotates a signal is sent to the calculator telling it to increment the total value by the input calibration value.
- I.1.1.6 <u>Timer</u> -- The timer can be programmed for up to two ON/OFF sequences for Monday through Friday operation. This means the sampler can be programmed to turn on and off twice, at the same time Monday through Friday. Saturday and Sunday programming can have only one ON/OFF sequence. However, Saturday and Sunday can have independent sequence times. Detailed information on timer programming can be found in Figures I.1.2 through I.1.5.
- I.1.1.7 Rotameter -- The rotameter gives a measurement of the flow. To achieve the PM® separation the actual flow should be 5 liters/minute.

#### I.1.2 PM10 Head

The PM₁₀ Head consists of three pieces, the cap made from a 2.5-inch PVC pipe cap, a 2.25-inch PVC tube connected with a 47mm Nuclepore screw cap, and the PM₁₀ separator made of Teflon®.

#### I.1.3 Filter Holders

The filter holders are 47mm Nuclepore Filter Holders with a 47mm under filter drain disk in each.

#### I.1.4 Battery Packs

The rechargeable battery packs are constructed from 2- to 6-inch PVC end

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s joined by a short piece of 6-inch PVC pipe. The batteries are two 6-volt unined in series and are recharged from a plug located on the side of the pack.

#### I.1.5 Sample Hanger

The sampler hanger is constructed of aluminum and when mounted will hold the sampler by the PVC caps of the handle assembly.

#### I.1.6 Sample Hanger

The sampler boxes will hold all of the equipment above except for the sampler hanger.

#### I.1.7 Miscellaneous Equipment

The following equipment is equally essential to conducting a saturation monitoring study.

Sampler Hanger Pole: commercial grade, telescoping, 8-foot, aluminum painter's extension pole

Chimney Clamp: TV antenna mounting brackets, two needed per sampler

Mounting Pipe: approximately 2 feet long

Tin Snips: for removal of mounts at end of study

Extension Ladder: 8 - 10 feet

Thermometer, Barometer

Wrenches, Socket Set

100-gm Tube Apiezon M Grease

2L n-Hexane: reagent grade

500-1000 mls Poly Squeeze Bottle

100 mls Dropper Bottle

Teflon® Forceps: non-serrated

Plastic Sandwich Bags

FIGURE 1.1.1

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# Saturation Monitor Circuit Board

AA BATTERY	
4 2 2 6	CALCULATOR: USED AS TOTALIZER OF FLOW
TIMER LOW BATTERY	
I/min AIR —— 10 LOW BATTERY LED	······································
POT #2  PULSE CIRCIUT  PUMP OFF TIME	7 8 9 %
	4 <u>5 6</u> X -
PULSE CIRCIUT 2 PUMP ON TIME 1 2 5 SWITCH #1: ON	0 - =
	POT #4 FLOW ADJUST
TUBING	

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FIGURE 1.1.2

## **TIMER CONTROLS**

#### **CONTROL BUTTONS**

1

2

3

4

) |

.O.



MO TU WE TH FR SA SU

ON STY 13:45

## TIMER CONTROL BUTTONS

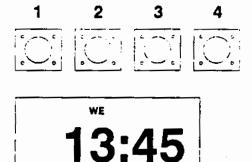
1= Low Battery Reset

2= Manual On/Off

3= Set Day/Time

4= Function

2 & 4= Reset Program



#### **SETTING THE TIME**

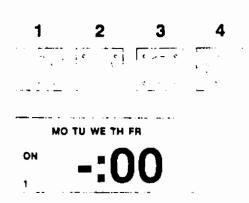
- 1- Press button #4 once
- 2- Press button #3 until current day shows in timer window
- 3- Press button #4 once
- 4- Hour will flash in timer window Press button #3 for desired hour
- 5- Press button #4 once
- 6- Minutes will flash in timer window Press button #3 for desired minutes

FIGURE 1.1.3

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# PROGRAMING WEEKDAY OPERATION

### SET PROGRAM START TIME



- 1- Press button #4 once
- 2- Hour will flash in timer window

  Press button #3 for desired start hour
- 3- Press button #4 once
- 4- Minutes will flash in timer window Press button #3 for desired minutes

### **SET PROGRAM END TIME**

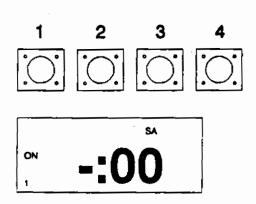
1	2	3	4
·- · - ·			
MO TU WE TH FR			
STY	-:0	)	
1 2			_

- 1- Press button #4 once
- 2- Hour will flash in timer window
  Press button #3 for desired end hour
- 3- Press button #4 once
- 4- Minutes will flash in timer window Press button #3 for desired minutes

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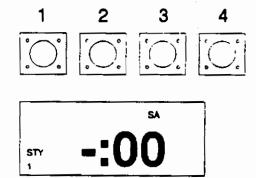
#### FIGURE I.1.4

## **Saturday Program**



# SET PROGRAM START TIME SATURDAY OPERATION

- 1- Press button #4 once
- 2- Hour will flash in timer window
  Press button #3 for desired start hour
- 3- Press button #4 once to enter
- 4- Minutes will flash in timer window Press button #3 for desired minutes
- 5- Press button #4 once to enter and advance to next program

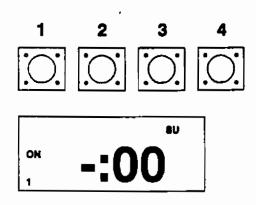


# SET PROGRAM END TIME SATURDAY OPERATION

- 1- Press button #4 once
- 2- Hour will flash in timer window Press button #3 for desired end hour
- 3- Press button #4 once to enter
- 4- Minutes will flash in timer window Press button #3 for desired minutes
- 5- Press button #4 once to enter and advance to next program

FIGURE 1.1.5

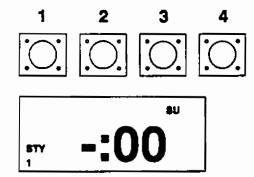
## **Sunday Program**



# SET PROGRAM START TIME SUNDAY OPERATION

- 1- Press button #4 once
- 2- Hour will flash in timer window

  Press button #3 for desired start hour
- 3- Press button #4 once to enter
- 4- Minutes will flash in timer window Press button #3 for desired minutes
- 5- Press button #4 once to enter and advance to next program



# SET PROGRAM END TIME SUNDAY OPERATION

- 1- Press button #4 once
- 2- Hour will flash in timer window

  Press button #3 for desired end hour
- 3- Press button #4 once to enter
- 4- Minutes will flash in timer window Press button #3 for desired minutes
- 5- Press button #4 once to enter and advance to next program

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#### 1.2 SAMPLING PROCEDURE

#### I.2.1 General

This sampling technique does not comply with EPA's ambient air monitoring regulations for  $PM_{10}$  sampling. The method has been shown to represent effective and efficient means for the collection of quality  $PM_{10}$  data. All sampling information should be recorded on data sheets as shown in Figure I.2.1.

#### I.2.1 Siting Requirements

There should be general conformance to 40 CFR, Part 58, Appendix E regarding probe criteris. The portable PM₁₀ sampler, should be positioned with the intake upward and located in an unobstructed area at least 30 cm from any obstacle to air flow. Accessibility to the units under all weather conditions, security of the monitoring personnel and equipment, should be considered when deploying the samplers.

#### I.2.3 Filter Media Inspection

All filters must be visually inspected for defects before initial weighing. Defective filters must be rejected. Batches of filters containing numerous defects should be returned to the supplier. The following are specific defects to look for:

- Pinhole a small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.
- Loose Material any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.
- Discoloration any visible discoloration that might be evidence of a contaminant.
- Filter Nonuniformity any obvious nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.
- Other a filter with any imperfections not described above, such as irregular surfaces or other results of poor filter construction.

  (Note: Quartz filters are susceptible to sloughing at the edges as a result of mechanical handling.)

#### I.2.4 Filter Preparation

Filters should be kept in protective cassettes and the unexposed filters must never be folded. The analytical laboratory (and/or filter manufacturer) will give each filter an ID number, which will be marked on two labels and attached to the filter cassette.

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#### 1.2.5 Sample Preparation

- I.2.5.1 <u>Impactor Maintenance</u> -- At every 7th sample, the impaction stage should be cleaned and regreased under a laboratory fume hood (preferably) or any well ventilated area (even on-site) as follows.
  - Disconnect the impactor section from the filter assembly and remove the rain cap.
  - Pushing with thumbs from the bottom, remove the impactor.
  - Rinse the impactor from top to bottom with hexane using the squeeze bottle, with particular attention to the impaction stage with concentric, grooved rings. Allow the impactor air-dry.
  - Prepare a mixture of hexane and Apiezon M grease (1/2-inch length grease to 100 mls hexane) in a dropper bottle. Vigorously shake the mixture until free from grease globs and is of a fluid consistency.
  - Put a large drop or two of the hexane grease mixture (cloudy solution) on the impaction stage. The drop should flow easily to the edge of the stage.
  - Inspect o-rings on impactor for fitness and replace if necessary. Carefully reinsert impactor from the top until top of the nozzle is flush with the top of the tube.
- 1.2.5.2 <u>Battery Pack Preparation</u> -- The voltage on each recharged battery pack should be checked with a volt-ohm meter (VOM) prior to load out:
  - Insert the VOM probe into the green socket and in turn, each of the two white pickups. The voltage reading should fall nominally around 12 volts.
  - If the voltage reading is less than 12 volts, take battery pack apart and look for loose connections. Sometimes, the fittings to the cover become loose and can be fixed by inserting a starred compression washer into the connection.
- I.2.5.3 <u>Charging Battery Pack on Sampler</u> -- Place charged battery pack beside sampler. Unclamp two side clips at base of sampler.
  - Lift sampler off used battery pack and place sampler on charged battery pack. Note: There are three pins on the bottom of the sampler. The pin closest to a side clip goes to the green receptacle on the battery pack.
  - Reclamp the two side clips. (Care must be exercised in transporting the battery packs to avoid jostling and subsequent cracking of plastic charging jacks.)

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#### I.2.6 <u>Sampling Procedures</u>

I.2.6.1 <u>General</u> -- After the sampling system has been assembled and calibrated, it can be used to collect air samples. Place sampler on a firm level surface.

#### I.2.6.2 Attach a Clean Filter

- Remove the outside plastic bag from the double-bagged clean filter assembly in the transport case. Place bag in an area where it will remain dry.
- Attach clean filter assembly at the quick-disconnect. Remove the plastic bag and quickly screw the assembly ring of the impactor section back onto the top of the clean filter assembly.
- Record the number of the clean filter assembly on the PM₁₀ Saturation Monitoring Data Sheet (Figure 1). Label filter base with ID number for filter.

#### I.2.6.3 Prepare Sampler for Run

- Unscrew either cap of bail assembly bar and remove bail assembly.
- Lift pump and timer assembly out by the top cap and support the
  mounting board on the edge of the sampler casing, taking care not to
  pull the connecting wires loose. Do not jar the pump hose fittings.
   Hold the top cap and do not grasp center electronic board.

#### I.2.6.4 Verify Sampler Operation Sequence

- Verify correct time of day, and week on timer LCD.
- To start pump and obtain the beginning flow rate, key the "on" button on timer pad to start pump.
- Determine if air is moving through the filter assembly by removing the rain cap and placing hand (or heavy mil plastic bag) over the flow nozzle. The rotameter will take up to two minutes to drop to zero. If the rotameter reads less than a couple hundred ml/min this is adequate. The flow will take the path of least resistance and will not seek small vacuum leaks. If a major leak is detected, check all filter holder joints and hose connections. Verify that filter canister is not leaking air from below the filter.
- Set flow rate on rotameter to the nearest 0.1 L/min, reading from the center of the ball, adjust for 5.0 L/min. The rotameter should be in the vertical position.

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Note: If a rotameter indicates zero or a very low reading, check the quick disconnect to be sure that the filter assembly is completely connected.

- Key the "off" button on timer pad to stop pump.
- Program the electronic timer for sampling times. See Appendix I.1.1.6, for instructions on timer programming.
- Input the liters/pulse calibration figure into the calculator.
- Place pump and timer assembly back into sampler body. Line up holes for the bail assembly bar and replaces bail assembly. Make appropriate entries on the PM₁₀ Data Sheet.
- Place sampler on mounting bracket.
- Position sampler using an extension hook. Hook bail and raise unit
  as vertically as possible. Standing away from sampler and raising
  at an angle will cause the hook to hit (breaking or jarring) the
  sampling inlet.

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#### FIGURE 1.2.1

# Saturation Monitoring Data Sheet

Operator:/ 90				Temperature: Pressure:				
Site No.	Sampler No.	Filter No.	Time of Day	Start Flow Tirr	ner Fior	End w Timer	Time of Day	Total Flow
1	Comment	B:	:	:		:	·	
2	Comment	B:	:	:		:	:	
3	Comment	<b>:</b>	:			:	:	
4	Comment	] 	:	:		:	:	
5	Comment		:	·				
6	Comment			:			:	
7	Comments	<b>3</b>		:		:*	:	
8	Comments	<b>:</b>		:		:	:	
9	Comments	<b>:</b>	•				:	
10	Comments	:						
11	Comment	<u></u>	<b>:</b>	:		. : . <b>.</b>	:	
12	Commente		·			·		
13	Comments	) <b>:</b>	i			•	:	
14	Comments					: :	:	
15	Comments		:				: ]	

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#### 1.3 SAMPLE HANDLING PROCEDURE

#### I.3.1 General

As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Potential for sample mass losses due to particle loss and/or volatilization, and filter damage increases if the filter is left in the sampler for extended periods.

• Record on the Data Sheet the average ambient temperature for the day and average barometric pressure for the day.

Note: Temperature and Pressure readings may be measured (recorded or estimated) on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected for sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If temperature and pressure readings are not available, seasonal average temperature and barometric pressure may be substituted. Care must be taken that the actual conditions at the site can be reasonably represented by such averages. It is therefore recommended that seasonal values represent actual values within 20°C and 40 mm Hg.

• Take sampler down from mount using extension hook. Positioned from directly under sampler, hook bale and lower away as vertically as possible. (This vertical take-away is critical as hook may otherwise dislodge rain cap or damage sampler head.)

#### I.3.2 Flow Rate Check

- Lift out pump and time assembly as described in Sampler Operation Sequence above.
- To get the ending flow rate, key the "on" button on timer pad to start pump.
- Record the flow rate from the rotameter to the nearest 0.10 L/min (read from center of ball) on the Data Sheet.
- Key the "off" button on timer pad to stop pump.

#### I.3.3 Exchanging New Filter for Exposed Filter

- Remove the outside plastic bag from the double-bagged clean filter assembly in the transport case, and place bag where it will stay dry.
- Unscrew the assembly ring of the top impactor section from the exposed filter assembly and immediately put the dry bag over the face of the filter assembly to keep it dry.

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- Unscrew the assembly ring from the top of the clean filter assembly and screw it onto the exposed filter assembly over the bag, to anchor the bag.
- Remove the entire exposed filter assembly at the quick-disconnect and place the exposed filter assembly into the transport case.
- Attach the clean filter assembly at the quick-disconnect (with the inside plastic bag still in place). Then remove the plastic bag and quickly screw the assembly ring of the impactor section back onto the top of the clean filter assembly.
- Record the number of the clean filter assembly on the data sheet.

  Label the filter ID number at the filter base.

#### I.3.4 Exposed Filter Instruction

- Back in the laboratory, unscrew the retaining cap from the top of the exposed filter assembly and discard the plastic bag.
- Locate the petri dish with the filter number which matches the number on the side of the filter base. (This should be the original petri dish in which the filter came.
- Unscrew the multi-holder adaptor from the filter assembly. Lift off the anti-twist ring from the base.
- Using forceps, carefully remove exposed filter from support grid, place filter back into its original petri dish, and replace the lid. (Be sure that drain disk remains in place on support grid.)
- Remove old ID label from filter base. Recheck this number to be sure it matches the number on the petri dish, and then discard tag.

#### I.3.5 Sample Preservation

- Completely log all data onto field data sheet and filter cassette immediately on return from sample retrieval.
- Place data sheets and cassettes into a separate container for each sampling date and seal with custody tape.
- With indelible ink marker, label container with date of sample run and number of samples enclosed.
- Maintain sample container(s) in refrigerator or freezer (less than 4°C is optimal). If unit is not lockable, seal door with custody tape.
- Do not remove until shipment.

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#### I.3.6 Sample Shipping (If Necessary)

- Ship samples weekly or biweekly, particularly if samples are suspected to be woodstove-impacted, or as directed by the project officer.
- Contact laboratory to ensure that someone will be available to receive shipment and store/analyze samples immediately upon receipt.
- Remove sample containers from refrigerator, place in garbage bags and twist-tie close.
- Immediately place in shipping container, packing sample containers firmly in packing material and ice packs (double-wrapped in garbage bags) so as to immobilize and to prevent jostling.
- Prepare a packing list, logging information on sample date and number of samples onto list, initial, seal in envelope put in plastics bag, and place with sample cartons.
- Seal container with strapping or plastic-coated tape and send via next-day parcel service.

#### I.3.7 Sample Receipt

The laboratory will provide the results (exposed filter weights) on all exposed filters within two weeks of receipt. All exposed filters shall be placed in a refrigerator or freezer before and after weighing when not undergoing equilibration for weighing.

#### I.3.8 Field Sample Custody

The operator who starts the sampler should record on the Data Sheets (Figure I.2.1): site designation and location, sampler serial number, filter ID number, sample date, average temperature, average pressure, initial flow rate, unusual conditions that may affect the results, and operators signature.

The operator who removes the samples should record on the data sheet: elapsed time of the sample run, final temperature, pressure, flow rate and existing conditions that may affect the results, explanations for voided or questionable samples, and operator's signature.

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#### 1,4 CALIBRATION PROCEDURES

To continually verify that the sampler flow rate operates close to the inlet design flow rate, average temperature and pressure measurements for each sample period are used. If these cannot be obtained, then seasonal average barometric pressure and temperature for the site may be substituted to determine the sampler flow rate. However, seasonal averages can be used only if the actual average temperatures and pressures at the site are not likely to deviate unreasonably from the seasonal average temperature and pressure (acceptable limits;  $\pm$  20°C and  $\pm$  40 mm Hg). The calibration procedure for the sampler yields calibrated flow rates at EPA-standard conditions versus ambient rotameter flow rates. Although the actual volumetric flow rate of the sampler is employed during sampling (necessary for PM₁₀ fractionation to collect the desired particle sizes), the flow is converted to the EPA-equivalent standard volumetric flow rate for calculation of the PM₁₀ concentration in ug/std m³.

Originally, each of the portable PM₁₀ sampling units were calibrated against a laminar flow element (LFE) (or NBS-traceable electronic or soap bubble flowmeter) by Lane Regional Air Pollution Authority (LRAPA), Springfield, Oregon. Six rotameter flow rates, ranging from _3.50 to 5.80 L/min, were calibrated against an LFE under specified temperature and pressure conditions.

To convert the calibration curve to ambient (i.e., field sampling) conditions, obtain LRAPA calibration forms for each of the samplers according to sampler ID number. Use the following equations to obtain a calibration curve with six data points. To obtain  $Q_{\rm std}$  (flow rate in L/min at EPA standard conditions; 760 mm Hg and 298 K), use the  $Q_{\rm std}$  values already provided by LRAPA for each LFE flow. As a check, Equation 1 provides the conversion for each flow rate determined at the LRAPA calibrated flow conditions to EPA-standard conditions.

Equation 1. Convert LRAPA Calibrated Flow Rates to Flow Rates at EPA STP Conditions.

$$Q_{std} \sim Q_c (P_c/P_{std})(T_{std}/T_c)$$

where  $Q_{\text{std}}$  = flow rate at EPA standard conditions in L/min (on LRAPA form)

P_{std} - 760 mm Hg

 $T_{\text{std}} = 298 \text{ K}$ 

Q_c - LRAPA LFE ambient calibration flow rate, in L/min

P_c - LRAPA ambient barometric pressure during calibration, in mm of Hg

T_c - LRAPA ambient temperature during calibration, in K

For each of the six  $Q_{\text{std}}$  values (corresponding to the six LFE ambient calibration flow rates) obtained using Equation 1, calculate the adjusted

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rotameter reading for use at field sampling conditions using Equation 2. This rotameter conversion corrects for the actual temperature and pressure at the site which are different from the LRAPA calibrated conditions. (Note: seasonal average temperature and pressure that fall within the given  $\pm~20^{\circ}$ C and  $\pm~40$  mm Hg acceptance limits may be used.) When adjusting or reading rotameter, take the rotameter reading at the center of the floating ball.

Equation 2. Obtain Corrected Rotameter Readings from LRAPA Indicated Rotameter Flow Rates.

$$I_{adj} - I_c [(P_c/P_{av})(T_{av}/T_c)]^{\frac{1}{4}}$$

where I_{adj} - corrected rotameter reading for T_a and P_a at the sampling site, based on the LRAPA calibration curve

I_c - LRAPA indicated rotameter flow rate corresponding to Q_{atd} (from LFE)

P_{av} - geographic average pressure at sampling site, in mm Hg (see Equation 3)

T_{av} = seasonal average temperature at sampling site, in degree K

Obtain  $I_{\text{adj}}$  for each  $I_{\text{c}}$  which corresponds to the flow rate,  $Q_{\text{atd}}$ , on the LRAPA calibration form.

Use Equation 3 only if the barometric pressure at the sampling site cannot be determined.

Equation 3. Determining Ambient Barometric Pressure from Elevation.

 $P_{x}$  - 760 - (elevation in meters x 0.076)

where Pa. - ambient barometric pressure in mm Hg

To obtain an ambient calibration curve for actual rotameter and flow rate (y-axis,  $I_{adj}$  versus x-axis,  $Q_{atd}$ ), plot the six data points for each sampling unit. Calculate the linear regression to find the slope (m), intercept (b) and the correlation of fit (r) for the sampling unit at ambient conditions from Equation 4.

Equation 4. Ambient Calibration Curve.

$$I_{adj} = m (Q_{atd}) + b$$

For sampling purposes, the portable PM-10 units must be operated at an actual flow rate of 5 L/min. Actual flow rates are required in order for the portable PM-10 to properly collect correct sized particles. However, flow rates at EPA standard conditions are used for the ambient calibration curve. To convert the actual 5 L/min to the corresponding  $Q_{\rm atd}$  flow value and thereby

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obtain the actual rotameter setting from the curve, use Equation 5.

Equation 5. Converting Actual Flow Rate to Flow Rate at EPA STP Conditions.

$$Q_{atd} = Q_a (P_a/P_{atd})(T_{atd}/T_a)$$

where Q = 5 L/min flow rate at ambient conditions

Calculate the  $Q_{\rm std}$  value for this actual 5 L/min flow rate. From the calibration curve at ambient conditions (or from the linear regression values), determine the corrected rotameter reading,  $I_{\rm adj}$  for  $Q_{\rm std}$ . Use this rotameter value  $\pm$  15 percent for the acceptable operating range for the sampler. (The warning limit range is  $\pm$  10 percent.)

Use a soap-bubble meter or LFE to verify rotameter reading. The rotameter reading should correspond to an actual flow rate of  $5\pm0.5$  L/min for warning limits and  $\pm0.75$  L/min for acceptable limits. (Note: the flow meters are used only as flow checks as the rotameters should be adjusted in accordance with the calibration curve.)

Liter/Pump-Pulse Factor - The "liters/pump-pulse factor" can be determined by setting the sampler to operate to a known rate. The ambient rotameter set point is convenient. Enter 'l' into the memory register of the calculator and start the sampler. In this way, the totalizer simply counts the number of pulses. Using a stop watch, measure the time it takes for a given number of pulses to occur; 50 to 60 pulses are reasonable - the elapsed time for 50 pulses is less than a minute. The equation below shows the calculation.

PROCEDURES
REGIONAL TECHNICAL SUPPORT
FOR CRIMINAL INVESTIGATIONS
EPA, REGION IV, ESD/NEIC

## PROCEDURES REGIONAL TECHNICAL SUPPORT FOR CRIMINAL INVESTIGATIONS EPA, REGION IV, ESD/NEIC

The attached Procedures are approved as the official mechanism by which the Region IV, Environmental Services Division shall provide technical support to the Atlanta Area Office of the National Enforcement Investigations Center's Office of Criminal Investigations.

James H. Finger, Director Environmental Services Division

Approved

Data

Bruce Mirkin, Special Agent-In-Charge Atlanta Area Office Office of Criminal Investigations National Enforcement Investigations Center

Approved

1/2/86

#### **PROCEDURES**

## Regional Technical Support for Criminal Investigations EPA, Region IV, ESD/NEIC

The Environmental Services Division (ESD) provides technical support to the Atlanta Area Office, Office of Criminal Investigations (OCI), National Enforcement Investigations Center (NEIC) for those investigations in Region IV requiring the collection of samples and/or laboratory analyses. These procedures address the means by which technical support is requested by the Atlanta Area Office and provided by ESD. Detailed technical procedures will be referenced to the various Standard Operating Procedures and Quality Assurance Manuals utilized by ESD.

The primary objectives of ESD support for Atlanta Area Office investigations are to provide accurate, complete, admissable and defensible reports and data for case development and subsequent legal proceedings. To accomplish these objectives, ESD and the Atlanta Area Office have jointly agreed to follow these procedures.

#### Technical Assistance Required by the Area Office

The Atlanta Area Office under the direction of a Special Agent-in Charge (SAIC) conducts criminal investigations in Region IV pursuant to Federal environmental laws and regulations. The technical assistance requirements for criminal cases vary from investigation to investigation. To assist the SAIC in identifying the type and scope of technical assistance required, a Technical Coordinator position has been established in the Area Office. The Technical Coordinator's primary function is to provide technical advise to the SAIC and Case Agents. It is the responsibility of the Technical Coordinator to assure that requests for technical support are directed to the appropriate Regional Program and ESD offices. After a decision by the SAIC to initiate a criminal investigation, the Technical Coordinator will discuss the case with the ESD Director or his designated representative. The ESD director in consultation and with the concurrence of the Deputy Regional Administrator (DRA) will decide whether technical support will be provided by ESD. If the technical divisions at NEIC rather than ESD are to provide the support, the Technical Coordinator will make appropriate contacts to assure that all appropriate aspects of the case (e.g., field investigation, analytical support and report preparation) are addressed. The technical Coordinator will advise the SAIC of the progress of these cases, and the SAIC in turn will inform the appropriate Regional program offices.

Following a decision that technical support will be provided by ESD, the SAIC will prepare a written request addressed through the DRA to the ESD Director. Copies of the request will be routed to the appropriate Regional program offices.

Prior to the conduct of the investigation, the Technical Coordinator will discuss the case with ESD personnel, and will inform the Case Agent of any technical requirements that may influence project planning. Conversely, it is the responsibility of the Technical Coordinator to inform ESD personnel of the presence of any known situations that would pose health risks or otherwise interfere with their operations.

Subsequent to the field investigation, the Technical Coordinator in consultation with the Case Agent will discuss with ESD personnel the progress of scheduled analyses and the potential need for additional or more detailed analyses. Information regarding the need to alter work schedules so as to meet changing Grand Jury or Federal Court dates will be transmitted immediately from the Technical Coordinator to ESD. When work schedules must be delayed, the technical reasons for the delay will be communicated from the Technical Coordinator to the Case Agent.

Upon completion of analyses and report preparation by ESD, and, prior to presentation of the results to a Grand Jury or Federal Court, the Technical Coordinator will arrange for an audit and inventory of evidence and files in ESD possession. A copy of the inventory will be provided to the Case Agent, to be used in selecting and preparing trial exhibits.

#### Project Requests

All requests for ESD support for criminal investigations shall originate with the Area Office. Any information obtained by ESD staff regarding potential criminal activity shall be immediately referred to the Area Office. Direct requests to ESD from any other Federal agency, State agency or local agency shall be referred to the Area Office SAIC for appropriate action.

Prior to an official request from the Area Office to ESD, an informal contact shall be made with the ESD Director or the appropriate ESD Branch or Section Chief concerning the availability of resources and expertise necessary for providing the requested technical support. If ESD is able to provide the support, the SAIC shall prepare an official request to the Director, ESD through the DRA with copies to the appropriate ESD Branch or Section Chief. In routine criminal investigations when ESD's priorities prohibit an immediate response, the RA/DRA will determine an appropriate course of action. If the RA/DRA determines that providing the requested technical support will be in the best interest of the Region, the support will be provided and adjustments will be made to other ESD commitments. ESD's technical support may begin immediately after receiving concurrence from the RA/DRA. If regional support is not available, the support will be requested from the technical divisions at NEIF. These divisions will normally provide support whenever Region IV is unavailable or whenever

the technical support is more appropriately provided by NEIC, e.g., undercover operations, especially sensitive operations, or cases involving more than one region. The SAIC has the responsibility of notifying appropriate regional organizational units, e.g., Office Directors, Division Directors, etc., of potential or on-going investigations receiving assistance from ESD or the technical divisions at NEIC.

All emergency requests shall be handled expeditiously. In such instances, ESD may provide the requested assistance immediately upon being notified by the SAIC that verbal approval has been obtained from the DRA. However, all such requests shall be followed by a written request from the SAIC, through the DRA to the ESD Director.

#### Project Coordination

Once the decision has been made to provide technical support, the ESD Director or the Deputy Director shall assign the project to the appropriate Branch/Section for assignment of a project leader. The project leader will be responsible for coordinating with the Case Agent to obtain necessary background information to determine logistical requirements, skill needs, laboratory support, etc. The project leader will coordinate all necessary activities with ORC, DOJ, FBI, trial attorney, etc. at the direction of the Case Agent.

The project leader shall discuss the technical and workload requirements with his/her immediate supervisor. A core team shall then be selected which will eventually conduct the study and/or coordinate analytical support. When required, additional staff will be assigned from other Sections or Branches with the approval of the appropriate Branch Chief or ESD Director. Once a core team has been selected, initial planning for the investigation shall begin under the direction of the project leader and in concert with the Case Agent and with the prosecutors if they are already involved in the matter.

#### Project Planning

After the appropriate or available background material has been obtained, specific assignments will be given to each member of the core team for development of a draft study plan. Concurrently, the team leader shall discuss analytical requirements and timeframes with Analytical Support Branch personnel. The draft study plan shall be assembled under the direction of the project leader and submitted to the core team, appropriate management, and Case Agent for review and concurrence. The timeframe for receiving comments will depend upon the urgency of the investigation, but in no cases should exceed 10 working days. During

emergencies an investigation may be conducted without the preparation of a detailed study plan. However, during these situations, a memorandum shall be prepared by the project leader briefly describing the technical work to be accomplished and stating that the investigation will strictly conform to appropriate ESD Standard Operating Procedures and Quality Assurance Manuals.

A final study plan will be prepared once all appropriate comments are received by the project leader. In general, these study plans should contain the following elements. However, upon advice of the Case Agent, the content and format of any study plan may be substantially changed to meet the needs of the particular investigation.

- Introduction a brief history and statement of the problem to be investigated;
- Objectives a statement as to what the investigation is to accomplish, how the information is to be used, and what specific laws may have been violated;
- o Scope a definition of the limits of the study;
- o Time Schedule a statement outlining when the study will be conducted, analytical results will be available, the draft report will be written, and the final report will be completed;
- Study Procedures the specific plan to collect the required information (not field methodology);
- Methodology specific field techniques to be employed. A statement that the techniques in the Division's Standard Operating Procedures and Quality Assurance Manuals will be employed shall be included. The use of any techniques not included in the Standard Operating Procedures and Quality Assurance Manuals shall be thoroughly justified and must produce evidence which can withstand objections by the defense;
- Analytical Requirements an estimate of the number of samples to be collected, required analyses, and which laboratory(s) will analyze the samples;
- o logistics an estimate of manpower requirements and a general description of specific functions of project personnel, special equipment and vehicles to be used, use of mobile laboratories, how samples are to be transported to the laboratory, etc.;
- Safety Plan a safety contingency plan will be included; and
- A statement that document control and chain-of-custody procedures will be followed.

#### Field Investigation

The field investigation will be conducted under the direct supervision of the project leader and the general supervision of the Case Agent responsible for conducting the investigation. ESD will endeavor to meet all objectives set forth in the study plan and any on scene changes or additional activities requested by the Case Agent. The ESD project leader or the ESD Safety Officer shall have sole responsibility for enforcing the provisions of the safety plan. The study will be conducted conforming to the requirements and objectives of the study plan and appropriate Standard Operating Procedures and Quality Assurance Manuals. Any deviations from the study plan or the appropriate Standard Operating Procedures and Quality Assurance Manuals must be approved and documented by the project leader. The deviations must produce evidence which can withstand objections by the defense.

During the field study, the project leader or designee is responsible for seeing that all chain-of-custody and quality control procedures for sampling, flow monitoring, field analyses, recordkeeping, etc. are followed. The field personnel are, however, expected to understand and follow the chain-of-custody and quality control procedures relative to their assignments. Following completion of the field activities, the project leader or designee shall account for all field documentation, such as field logbooks, sample tags, and chain-of-custody records, and verify that they are complete. Sample tags will remain on the sample containers in the custody of the Analytical Support Branch until relinquishment to the court or anal disposition of the case.

#### Laboratory Support

A laboratory coordinator will be appointed by the Chief, Analytical Support Branch to represent the laboratory on the core team. The primary responsibilities of the laboratory coordinator are to assist in determining the analytical needs of the investigation and to provide overall coordination of sample analyses and data reporting.

Upon delivery of samples to the ESD laboratory, the samples shall be immediately transferred, via chain-of-custody procedures, from the project leader or designee to the laboratory sample custodian or designee. After receipt of samples, the sample custodian shall immediately transport the samples to the sample custody room. The sample custodian shall document the condition of the samples and verify the uniformity of information on the sample tags and chain-of-custody records prior to placing the samples in the sample custody room.

All sample handling, sample preparation, and analyses shall be in strict conformance with the Analytical Support Branch Standard Operating Procedures and Quality Assurance Manual.

The laboratory coordinator shall notify the project leader as results become available. The project leader will review these results with the core team including the Technical Coordinator and determine whether additional sampling and/or analyses are required to meet the objectives of the investigation.

Final analytical data shall be reported directly to the project leader after all QA/QC procedures have been completed. Any analytical problems or deviations concerning holding times, analytical procedures, etc. shall be reported to the project leader and the Case Agent. When requested by the Case Agent, this information will be documented in a memorandum stamped "CONFIDENTIAL" and transmitted to the project leader, Technical Coordinator, and Case Agent.

#### Final Report

The project leader is responsible for preparing a final investigative report. Input shall be provided by all appropriate members of the core team.

A draft report shall be prepared for internal review by all core team members and the Case Agent. The draft report may also be reviewed by other appropriate staff, i.e., supervisors, technical experts, ORC, etc., with the concurrence of the Case Agent. All draft reports shall be destroyed upon completion of a final report.

A final investigative report will be prepared by the project leader. This report shall contain factual information and observations but shall not contain conclusions, recommendations, or personal opinions. At the request of the Case Agent, a memorandum will be prepared containing conclusions, recommendations, or personal opinions. When this is done, the memorandum will be stamped "CONFIDENTIAL" on each page and either hand delivered or delivered via certified mail to the Case Agent. The final report(s) shall be delivered directly to the Case Agent who shall be responsible for ultimate distribution.

#### Document Control

The core team members are responsible for the initial collection and maintenance of all documents, records, and evidence generated during the investigation. As required by the project leader, but no later than the completion of the draft investigative report, all documents, records, and

evidence obtained during the field investigation shall be delivered to the project leader who shall immediately construct an inventory of all documents, records, and evidence obtained. A copy of the inventory will be furnished to the Case Agent. If requested, the project leader will deliver all such records to the Case Agent.

All original analytical data and supporting documentation, e.g., chromatograms, mass spectra, QA/QC records, calculations, etc shall be maintained by the Analytical Support Branch according to their Standard Operating Procedures and Quality Assurance Manual. If requested, copies of all records shall be provided to the Case Agent. Dissemination of such records shall only occur under Federal court order, as directed by DOJ, or as directed by the prosecuting attorney. The Laboratory Coordinator shall construct a project file of all laboratory data and supporting documentation immediately after completing analyses and reporting of data to the project leader. An inventory of that file will be prepared and furnished to the project leader and the Case Agent.

NEIC will support the construction of the inventory and assure the completion of the documentation through the evidence audit program prior to presentation of the results to a Grand Jury or Federal Court.

All documents, records, evidence, etc. retained in the Environmental Services Division will be maintained in a locked filing cabinet or a secure area under the direct control of the appropriate Branch or Section Chief.

#### Sample Disposal

All excess samples and/or sample containers shall be maintained in the sample custody room until written authorization for sample disposal is received from the Case Agent. Because of lack of space in the sample custody room, the Case Agent will expeditiously inform the Analytical Support Branch when samples can be disposed. Sample disposal procedures shall be as described in the Analytical Support Branch Standard Operating Procedures and Quality Assurance Manual.

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### APPENDIX K BRANCH SAFETY PROTOCOLS

#### K.1 GENERAL

#### K.1.1 Introduction

This SOP has attempted to make safety protocols an intrinsic part of the procedures for each specific operation addressed. However, there are some protocols which were either not included in the other sections of this document or which merit more emphasis than given as part of normal operational procedures.

It is the purpose of this appendix to address those protocols.

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#### K.2 HAZARDOUS WASTE SECTION SAFETY PROTOCOLS

#### K.2.1 Equipment

Section field employees will be provided with the following field clothing and safety equipment:

- Coveralls or long sleeve shirts and pants (3 pr)
- Rain suit
- Snow suit and ski mask
- Work gloves
- Safety glasses (prescription if necessary)
- Goggles
- Hearing protection
- Hard hat
- Steel toe/shank safety boots (leather and rubber)
- Air purifying respirator (APR)
- Flashlight (intrinsically safe)
- First Aid Kit
- Equipment bag

The Branch warehouse personnel will serve as custodian for all section field equipment. In this capacity, warehouse personnel will verify the proper operation of all field equipment prior to its being deployed for field use. Warehouse personnel will also be responsible for the upkeep of field equipment to include maintaining an adequate inventory of replacement parts and support items.

Hazardous Waste Section field personnel will be responsible for properly operating and maintaining equipment in the field. Should the equipment malfunction or be broken, field personnel are responsible for reporting the condition to the warehouse upon its return. The report will include as accurate a description or account of the problem as possible.

Under no circumstances will Hazardous Waste Section personnel operate equipment for which they have not received training or have insufficient familiarity to conduct safe operations.

The following activities will require a familiarization exercise for personnel prior to the actual execution of the work:

- Confined space entry
- Level A, B or C operations
- Drilling or power augering
- Drum openings
- Brush cutting with power equipment
- Boat operations
- Generator operation
- Steam cleaning

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#### K.2.2 Training Status Tracking System

The Hazardous Waste Section operates a computer system for tracking the status of required safety training for all personnel involved in hazardous waste field operations within the Environmental Services Division. The system tracks the following safety training:

- Medical monitoring physicals (Annual renewal)
- 40-hour hazardous waste training (No required renewal)
- 8-hour refresher training (Annual renewal)
- Cardio-pulmonary resuscitation (CPR) certification (Annual renewal)
- First aid certification (Tri-annual renewal)
- Fit testing (Annual renewal)

It is the responsibility of the Hazardous Waste Section safety officer (or a designee) to notify field personnel (or their supervisor) when renewals of required training are due. The notification will be by letter and will be at least 60 days prior to the actual renewal date. Scheduling training will be the responsibility of the each individual unless otherwise stipulated in the notification. Upon scheduling of the training, the individual will notify the Hazardous Waste Section safety officer/designee of the date. Upon successful completion of training, a copy of the certificate received will be sent by the individual to the safety officer/designee for inclusion in their safety training file.

In the event a field person's required training expires, the individual will not be allowed to enter onto hazardous waste sites. The individual and their supervisor will be notified by letter of the change in status. Upon successful completion of the required training, the individual and their supervisor will be notified by letter of their return to prior status.

#### K.2.3 Specific Site Safety Plans

A site specific safety plan will be developed for every hazardous waste site project conducted by ESD. The plan will use the form included in this section. The plan will be typed and submitted to the Hazardous Waste Section Safety Officer and the Occupational Health and Safety Officer (OHSD) for approval. Prior to commencing site activities, field personnel will be briefed on the contents of the safety plan. The plan's emergency instructions and directions will be posted in a conspicuous location at the site command post. When there is more than one organization involved at the site, the development of the safety plan should be coordinated among the various groups.

#### K.2.3.1 Augering and Drilling Operations

K.2.3.1.1 <u>Underground Utilities</u> -- All underground utilities must be located prior to commencement of drilling operations involving the drill rig and power augers. Complete the underground utilities checklist presented in Figure APP K.2.2 and prepare a site map showing the locations of all underground utilities identified.

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K.2.3.1.2 Above Ground Utilities -- All above ground utilities must be located prior to commencing drilling/auguring activities. A map will be prepared showing the locations of all power lines, telephone lines, video cables, guy wires, and other objects which could pose a hazard to personnel operating the drill rig, power auger, or hand auger with multiple extensions. The site safety officer will insure that all operations are kept well clear of such hazards.

#### K.2.4 Site Operations

- K.2.4.1 <u>Initial Site Survey and Reconnaissance</u> -- The purpose of an initial site survey/reconnaissance is to accomplish one or both of the following objectives:
  - Determine the hazards that may exist which could affect site personnel.
  - Verify existing information or obtain new information about the site.

To accomplish the first objective listed above, an assessment of the real or potential dangers from fire, explosion, airborne contaminants, radiation, and oxygen deficient atmospheres must be made. This assessment will be made as follows:

- Combustible Gases -- The atmosphere in any location capable of containing or generating a combustible concentration of gases will be monitored with a combustible gas meter. Any response of the meter in excess of 25% of the lower explosive limit (LEL) will cause an immediate evacuation of the site.
- Oxygen Deficiency -- Any location capable of containing or generating an oxygen deficiency either by depletion or displacement will be monitored with an oxygen meter. Any reading less than 19.5% oxygen will result in the use of self contained breathing apparatus (SCBA).
- Organic Vapors and Gases -- The atmosphere will be monitored with both a photoionization detector (PID) and a flame ionization detector (FID). Any response above background concentrations will cause an upgrade to level C respiratory protection. Any response above 5 ppm when contaminants are not known, will cause an upgrade to level B respiratory protection. A response above 200 ppm when contaminants are not known will cause an upgrade to level A protection.
- Inorganic Vapors and Gases -- The number of direct reading instruments with the capability to detect and quantify non-specific inorganic vapors and gases is extremely limited. PIDs have a very limited capability in this area. If specific inorganics are known or suspected of being present, an attempt should be made to provide appropriate monitoring if possible. In the absence of a monitoring capability always assume a worse case scenario.

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• Radiation -- A radiation survey will be conducted of the site. The primary survey instrument will be a Geiger-Mueller detector for beta/gamma radiation (see Appendix G for a discussion of limitations). Any response above background will result in evacuation of the area where elevated readings are obtained.

Following the initial survey, monitoring will be repeated when new areas of the site are entered, or when operations likely to cause a release are being conducted.

- K.2.4.2. <u>Stress</u> -- Field personnel on hazardous waste sites are exposed to both psychological and physiological stress. Psychological stress is countered with adequate training and job proficiency. Physiological stress is primarily due to exposure of the worker to extremes of heat and cold.
  - Heat Stress -- Heat stress can be the result of working during hot weather or wearing protective clothing that inhibits natural ventilation. It can occur even under moderate temperature condition. The following protocols are to be used to counter heat stress.

To allow workers to replace body fluids lost, water will be available at the site. Gator-Aid® will be available at the discretion of the site safety officer.

Cooling vests will be made available. Their use may be made mandatory at the discretion of the site safety officer.

At the discretion of the site safety officer, workers' vital signs will be monitored (i.e., body temperature, blood pressure and heart rate). If deemed necessary, workers will be fitted with heat stress monitors.

Adequate shade will be provided to shelter workers from direct exposure to the sun during rest periods.

Work teams will be rotated so that individual time on especially stressful jobs is minimized.

Field personnel are to be encouraged to maintain their physical fitness.

Intake of diuretics (coffee or alcohol) is to be minimized prior to field work.

<u>Cold Stress</u> -- Exposure to extreme cold can result in hypothermia. Field work during periods of low temperatures and wind should be conducted to minimize the possibility of hypothermia. The following protocols are to be followed:

Workers will dress as warmly as possible using the principle of layering their clothing to maximize protection.

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Careful attention will be used to wearing gloves when handling metal equipment.

At the discretion of the safety officer, work tours will be limited to minimize exposure to the cold.

Warm shelter will be made available for workers during breaks. The use of vehicles for warm shelter is discouraged due to the possibility of carbon monoxide exposure.

The safety officer will carefully observe workers for signs of hypothermia/frostbite.

K.2.4.3 <u>Site Control</u> -- Site control serves to minimize exposure to contaminants. This is accomplished in the following ways:

Provide site security to exclude unnecessary personnel.

Limit the number of workers and equipment on-site to the minimum required for effective operations.

Conduct operations to reduce personal exposure and minimize the potential for airborne dispersion.

Implement decontamination procedures.

- K.2.4.3.1 <u>Work Zones</u> -- In order to control access of personnel and equipment to possible contaminants, the site will be divided into work zones. There will be three categories of zones utilized.
  - 1. Support Zone This is the outermost boundary of the site. In order to enter this zone, personnel must have current training required for hazardous waste site work. Contamination of personnel and equipment in this area is unlikely.
  - 2. Exclusion Zone This is an area within the support zone, where actual operations are being conducted. Access to this area is limited to personnel and equipment being utilized at that particular time. The risk of contamination in this area is high.
  - 3. Contamination Reduction Zone This area serves as a corridor between the exclusion zone and the support zone. All personnel and equipment passing through this corridor from the exclusion zone to the support zone must undergo appropriate decontamination.
- K.2.4.3.2 <u>Decontamination</u> -- All equipment and personnel will undergo an appropriate decontamination prior to entering the support zone. Under no circumstances will contaminants be knowingly transported off a site except when properly containerized as samples or waste solvents. All disposable equipment will be properly bagged for disposal prior to entering the support zone.

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#### FIGURE APP K.2.1 SAFETY PLAN

	SAFETY PLAN							
Site Name: Contact:								
Address:								
Phone Number:		<del>''</del>						
Purpose of Site Visit: Proposed Date(s) of Work: Directions to Site:								
Site Investigation Team:								
Personnel*	Safety Category	Responsibilities**						
·								
		·						
* All employees have been to 29 CFR 1910.12 requirement	rained/medically monitor							
** Note: Site Safety Office:	: Designee.							
Plan Preparation								
Prepared but		Date						
Prepared by:								
Site Status:	Active, Inac	tive, Unknown						
EMERGENCY INFORMATION:								
Local Resources:	·							
Ambulance (Name):		Phone:						
Hospital (Name):		Phone :						
Police (Local or Stat	:e):	Phone :						
Fire Department:		Phone:						

#### FIGURE APP K.2.1 (Con't)

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#### Office Resources:

ESD Office Phone: 404/546-3351 EPA - Emergency Response - Atlanta Phone: 404/347-4062

HWS Chief - William R. Bokey Work: 404/546-3300 Home: 549-2611 RCRA Unit Chief - Bill Cosgrove Work: 404/546-3321 Home: 742-7331 Superfund Unit Chief - Steve Hall Work: 404/546-3173 Home: 548-7600 Safety - Jim Gray Work: 404/546-3308 Home: 543-0710

#### EMERGENCY CONTACTS:

Poison Control Center Phone: 800/282-5846
National Response Center Phone: 800/424-8802
(FOR ENVIRONMENTAL EMERGENCY ONLY)

Directions to Hospital (Attach Map if Available):

SAFETY AND HEALTH RISK ANALYSIS

Vaste Types/Chemicals:

Hazard Evaluation:

Known or Suspected Hazardous/Toxic Materials (If applicable include: PEL/IDLH and/or TLV-TWA/TLV-STEL, LEL, flammability, odor, reactivity, stability, corrosivity)

Overall Hazard:

Serious

Low

Unknown

FIGURE APP K.2.1 (CON'T)

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Site Perimeter Establishment:
Map/Sketch attached?  Perimeter identified?  Zone(s) of contamination identified?
Recommended Level(s) of Protection:  A B C D
• Level of Protection: (check those that apply)
• Modifications: Respiratory:
Field Dress:
Monitoring Procedures/Equipment*:
HNU OVA Radiation Survey Meter Explosimeters Oxygen Meter Other, Specify * All instruments are calibrated in accordance with the Engineering Support Branch Standard Operating Procedures and Ouality Control Assurance Manual or by the manufactures specification.
Method of Air Surveillance:
Additional Site Specific Information/Stipulations:
Site Decontamination Procedures:
Confined Space Entry (check one): YES NO
If yes, define procedures to be used:

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## FIGURE APP K.2.2 UNDERGROUND UTILITIES CHECKLIST

Utility	Locator/Contact Person	Phone #	Date of Location
Power			-
Telephone *			
Gas	·		
Water			
Sewer			
Other			

IMPORTANT: Check all proposed drilling locations with a pipe-seeker. As a minimum, the first four feet of a power bored hole will be dug using a post hole digger/hand auger. Personnel involved in the drilling will wear eye protection in addition to normal safety gear, appropriate for the required level of protection. The site safety officer will insure that all personnel remove watches, rings and other jewelry, as well as securing loose fitting or dangling articles of clothing while in the vicinity of the drilling operations. Additionally, the safety officer will insure that a 90 degree clear zone is maintained for a radius of at least 25 feet behind the drill rig.

^{*} Include non-AT&T lines such as Sprint, MCI, etc.

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#### K.3 AIR AND WATER COMPLIANCE SECTION SAFETY PROTOCOLS

#### K.3.1 Equipment

- K.3.1.1 <u>General</u> -- Section field employees will be provided with the following personal field clothing and safety equipment:
  - Coveralls and/or long or short sleeve shirts and pants (three pairs)
  - Rain Suit
  - Safety Glasses
  - Hard Hat
  - Steel Toe/shank safety boots (leather and rubber)
  - Individually fitted full face respirators

Personnel required to work on a hazardous waste site will be provided with any and all other safety equipment and clothing provided to the Hazardous Waste Section staff (Appendix K.2).

- K.3.1.2 <u>Water Compliance Unit</u> -- All the field equipment used by the Water Compliance Unit (WCU) is stored and maintained by Hazardous Waste Section (HWS) personnel at the Division warehouse. As in Section K.2.1, HWS personnel are responsible for the upkeep of WCU equipment. WCU field personnel are responsible for properly operating and maintaining equipment in the field.
- K.3.1.3 <u>Air Compliance Unit</u> -- Monitors, sampling equipment, delicate lectronic instruments and other equipment used by the Air Compliance Unit are stored and maintained by the ACU staff at the Air Laboratory. The ACU propane generator and some larger monitor housings are stored at the warehouse. The ACU staff is responsible for proper equipment operation and maintenance in and out of the field.

#### K.3.2 Training Status Tracking System

The HWS operates a computer system for tracking the status of required safety training for all ESD personnel involved with hazardous waste site field operations (Section K.2.2). All Air and Water Compliance Section (AWCS) personnel involved with hazardous waste site evaluations and are included in the HWS tracking system.

#### K.3.3 Specific Site Safety Plans

When conducting or assisting with the conduct of investigations or monitoring at hazardous waste sites the AWCS staff will follow the requirements of Section K.2.3. The AWCS staff is usually called in by the OSC or Emergency Response staff after the initial reconnaissance. Generally, the safety plan will be developed by the HWS staff before the AWCS staff is required to enter a site. Therefore, the AWCS staff will work under the safety plan in use. In the event the AWCS staff is first on site, the AWCS project leader will develop a safety plan as outlined in Section K.2. to comply with safety category requirements and o protect AWCS personnel.

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Routine ACU ambient and air toxics monitoring and WCU compliance inspections and investigations will not require a site specific safety plan beyond the standard safety precautions required and used on all field activities.

#### K.3.4 Site Operations

- K.3.4.1 <u>Water Compliance Unit</u> -- Wastewater and its by-products pose potential hazards. The hazards include the waterborne diseases such as typhoid fever, paratyphoid fever, dysentery, infectious jaundice, hepatitis, and the danger from tetanus. The best defense against infection is the practice of good personal hygiene. The following safety guides (1) should be observed whenever working around wastewater:
  - Hands and fingers should be kept from the nose, mouth, eyes, and ears.
  - Rubber gloves should be worn when handling wastewater, screening, sludge, or grit; or whenever handling equipment which has been in contact with untreated wastewater or sludge.
  - Gloves should always be worn when hands are chapped or burned or when the skin is broken for any cause.
  - Before eating and after work, the hands should be washed thoroughly with soap and hot water.
  - All cuts and scratches should be given immediate first aid.
- K.3.4.2 <u>Air Compliance Unit</u> -- A number of hazards exist for ACU operators conducting air monitoring or instrument calibrations. Many of the calibration gases that are used are toxic. The samplers are often in locations that can require climbing ladders. Samplers can pose an electrical shock hazard when wet. The following safety guidelines should be followed when conducting either air monitoring or instrument calibrations.
  - All calibration gases should be vented to the outside of air monitoring enclosures during calibration.
  - Care should be exercised while climbing ladders.
  - Non-conductive shoes should be worn on wet roofs when changing samples when the sampler is wet. Also make sure that all extension cord connectors are located in dry areas.
  - Hoods should be used when solvent rinsing equipment.
- K.3.4.3 <u>Safety Precautions for Municipal Wastewater Treatment Plants</u> There are many hazards in and around municipal wastewater treatment plants. In many WWTPs, open channel flow measurement systems are hazardous and are located in dangerous areas, such as upstream of large screw pumps or high flow open pit discharges. Walkways and ladders around the various treatment units may be unsafe. In many older WWTPs, there are no railings around treatment units and

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unibusys Heights can be a safety hagard for many above-ground treatment units. The following sets: / guidelines should be followed by all field personnel:

#### K.3.4.3.1 On-Site Precautions

- Field personnel should never enter an enclosed, below grade, or unventilated structure such as a manhole. Caution should be exercised when entering a pump room or lift station. When entering a below grade area, e.g., lift station, wet well, etc., an initial reconnaissance should be conducted as described in Section K.2.4.1 and adhere to guidelines in Appendix B. An emergency escape pack should accompany each person when entering a potentially oxygen deficient area.
- Proper safety shoes or boots should be worn to prevent slipping on wet surfaces.
- In WWTPs where the final outfall or open channel flow measurement system is hazardous, field personnel should tie off with a safety line or harness when taking wastewater samples or water depth measurements. In no cases should field personnel enter such areas without another person standing by.
- Field personnel should not attempt to access elevated areas which are hazardous or where heights may be a safety hazard, such as above ground treatment units with dangerous walkways.
- Field personnel should be cautious of moving mechanical equipment such as mechanical bar screens, grit scrapers, belt drives, and motors.

#### K.3.4.3.2 Sampling

- Field personnel should never conduct in-plant sampling or other inplant measurements without another person standing by.
- Field personnel should wear rubber surgical gloves during sample collection to prevent sample contamination and reduce exposure to infectious organisms.
- Field personnel should attach sample containers to a sampling pole when collecting samples from inaccessible locations or hazardous areas. Precautions should be taken to avoid overhead electrical lines and other hazards.
- K.3.4.4 <u>Safety Precautions at Industrial Facilities</u> -- Inspections conducted at industrial facilities require that the inspector adhere to the safety rules maintained at the industry for protection of visitors and facility personnel. The inspector should request from facility personnel clarification of safety precautions and the particular dangers that he might encountered at the facility. The inspector must not sign release waivers of liability that the incility may require prior to entry into the facility.

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#### K.3.5 Reference

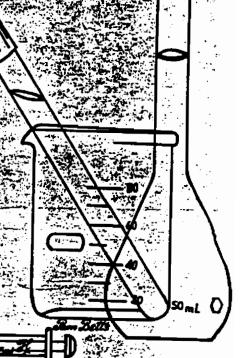
(1) Operations of Wastewater Treatment Plants, MOP/11, WPCF, 1976, p. 487.



# Laboratory Operations and

Quality Control
Manual

Region IV:
Athens, GA



# ANALYTICAL SUPPORT BRANCH OPERATIONS AND QUALITY CONTROL MANUAL

ENVIRONMENTAL PROTECTION AGENCY ENVIRONMENTAL SERVICES DIVISION REGION IV ATHENS, GA 30613

#### DISCLAIMER

The mention of trade names or commercial products in this manual is for illustration purposes, and does not constitute endorsement or recommendation for use by the Environmental Protection Agency.

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#### 1. INTRODUCTION

1.1. This manual is designed to delineate the routine operation of the USEPA Region IV Analytical Support Branch. The primary purpose of this document is to establish and maintain uniform operational and quality control guidance for regional chemical and microbiological analytical activities and regional laboratory evaluation, laboratory certification, contract laboratory performance monitoring and quality assurance activities. The establishment of, and adherence to, uniform elements of an intralaboratory quality control program are essential to the production of reliable analytical data.

- 1.2. Coordination of the overall regional quality assurance activities, and likewise the Analytical Support Branch quality assurance activities, rests primarily with the Regional Quality Assurance Officer (QAO). The functions and responsibilities of the QAO are identified in the Region IV Quality Assurance Program Plan. The QAO serves as a focal point for the dissemination of information and provides program managers with technical advice pertaining to the development, implementation, and operation of regional quality assurance activities. Liaison with the Office of Research and Development, Quality Assurance Management Staff (QAMS), is maintained by the QAO. Implementation of agency quality assurance policies applicable to the laboratory is the responsibility of the Chief, Analytical Support Branch.
- 1.3. The Branch has an individual responsibility for the operational aspects of the quality assurance activities. Assignments are made to insure involvement of all activities in quality assurance and to maintain control of quality assurance data.
- 1.4. This manual and the quality control protocols described are not to be viewed as all inclusive. Rather, they serve as a basic foundation on which to build a stronger quality assurance program within the Branch. Some quality assurance documents are included by reference
- 1.5. All ASB staff are required to familiarize themselves with the sections of this manual that pertain to their individual operations.

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#### 2. BRANCE ORGANIZATION AND OPERATION

- 2.1. The Analytical Support Branch organizational structure is shown in Figure 2-1.
- 2.2. The Branch is a technical support activity with the following functions:
  - 2.2.1. Provides chemical and microbiological laboratory services in support of all regional program needs.
  - 2.2.2. Provides consultation and assistance to local, State, and other agencies in matters of analytical methodology and laboratory quality assurance.
  - 2.2.3. Provides personnel as regional representatives to national programs relating to selection, validation, and promotion of the use of official EPA analytical methods.
  - 2.2.4. Participates in national and regional interlaboratory method evaluation studies.
  - 2.2.5. Provides laboratory evaluation and certification support for the region.

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### **FIGURES**

# BRANCH ORGANIZATION AND OPERATION

SECTION 2

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#### U. 8. ENVIRONMENTAL PROTECTION AGENCY ENVIRONMENTAL SERVICES DIVISION ANALYTICAL SUPPORT BRANCH

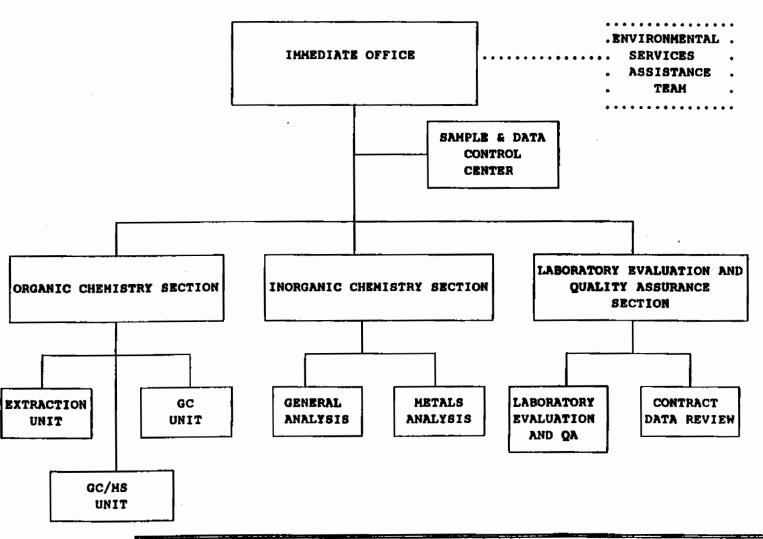


Figure "-1

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#### 3. LABORATORY CHAIN-OF-CUSTODY

#### 3.1. <u>Introduction</u>

3.1.1. Complete documentation of the sample collection and handling process is an extremely important aspect of a regulatory monitoring effort. Formal chain-of-custody procedures provide for a written record of sample traceability, accountability, and serve to validate sample integrity. All samples received by ASB for chemical analysis are controlled by these procedures. Field sample custody procedures are detailed in the Environmental Compliance Branch, Standard Operating Procedures and Quality Assurance Manual.

3.1.2. All custody documentation on samples for microbiological analyses will be maintained in the microbiology sample logbook by the project microbiologist. The same documentation will be maintained as listed for chemical samples. The sample custodian will not maintain these records.

#### 3.2. Sample Custody Forms

- 3.2.1. The following sample custody forms are shown in Form 3-1 through 3-3:
- c Chain-of-Custody Seal (Form 3-1).
- o Chain-of-Custody Record (Form 3-2).
- o Custody Room Sample Log (Form 3-3).
- 3.2.2. In addition to these forms, custody information is maintained in the master microbiology logbooks, Sample and Analysis Management System (SAAMS), computer sample log, the chemistry and microbiology field logbooks, and in the individual analytical data books.

#### 3.3. Standard Operating Procedure

- 3.3.1. All samples are received by the sample custodian or an alternate. At the time of the receipt, the custodian or alternate performs the following:
  - 3.3.1.1. Document whether the individual samples, boxes, or ice chests were sealed upon receipt; also document condition (if damaged) of sample container in "remarks" section of the custody form.
  - 3.3.1.2. Sign all chain-of-custody records, and identify the date and time of sample receipt.
  - 3.3.1.3. Log all samples into the SAAMS noting the following information: Sample log number, source of sample, project number, station description, if sealed yes or no, date and time of

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collection, who collected, date and time received, received by, received from, analysis required, and any other pertinent remarks. The computer log screen is shown in Figure 3-1.

- 3.3.1.4. Place sample numbers on all sample containers and secure samples in the secured-refrigerated custody room and/or secured refrigerators used for storing VOA samples.
- 3.3.1.5. After sample logging is completed, computer print-out sheets listing the required analyses for all samples will be given to each project chemist and placed in the project file. Computer data reporting sheets will be given to the appropriate project chemists.
- 3.3.1.6. A copy of the field custody form, along with a computer printout of the tests to be analyzed for, will be maintained in the ASB files. The original field custody form and a copy of the computer printout will be sent to the individual(s) responsible for sample collection.
- 3.3.2. For an analyst to receive samples for analysis, the following actions are required (access to the custody room by analysts requires approval by either the Organic Chemistry or Inorganic Chemistry Section Chief who issues them a key to the custody room):
  - 3.3.2.1. The analyst will fill out the Custody Room Sample Log Sheet (Form 3-3), listing the sample type and samples by individual SAD numbers, and initial the log.
  - 3.3.2.2. The analyst will remove the required samples from the custody room.
  - 3.3.2.3. The analyst is responsible for custody of samples during analysis. He/she will document date of analysis, type of analysis, log numbers, and signature in the data logbook.
  - 3.3.2.4. The analyst will return the samples to the custody room when he/she is finished with the analyses. In no case will the original samples remain outside the custody room during non-duty hours unless all analyses are complete and the sample is to be discarded. When the samples are returned, the analyst will again sign and date the Custody Room Sample Log Sheet.
  - 3.3.2.5. The Custody Room Sample Sheet will be maintained as a permanent file and entered into SAAMS.
  - 3.3.2.6. When the project chemist has been authorized to dispose of samples, he/she fills out the Custody Room Sample Log, checking disposal, listing the samples by type and SAD number, dating, and initialing. The serialized tags will be removed from the sample containers at the time of disposal and returned to the individual or group responsible for the collection of the samples.

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3.3.3. If samples are received at a field laboratory, or after normal business hours at the main laboratory, the following procedure will be allowed:

- 3.3.3.1. A designated individual will receive all samples by signing and dating all field sheets.
- 3.3.3.2. He/she will then open the ice chests, remove the sample containers, perform any required analyses, and split samples into additional sample containers if necessary.
- 3.3.3. He/she should then put all sample containers back into ice chests and either seal the chests or place in custody room.
- 3.3.3.4. The custodian will assume custody the next day, or when the samples arrive at the central laboratory from the field location.

#### 3.4. Audit of Custody Records

3.4.1. Audits of custody information will be performed by the Branch Chief or his designee. These audits will include an examination of custody documentation of randomly selected samples for: traceability, completeness, an accuracy. The results of these audits will monitor the general effectiveness of the custody procedures.

#### 3.5. Policy for Disposal of Laboratory Samples

- 3.5.1. No criminal investigation samples or sample containers will be disposed of until disposal has been authorized by the appropriate officer of the court.
  - 3.5.1.1. Water samples requiring BOD, BAC-T, Extractable Organics and Volatile Organic analyses may be disposed of when analyses are complete.
  - 3.5.1.2. All other samples (e.g., water, soil, sediment, tissue, waste, etc.) will be disposed of as follows:
- 3.5.2. They will be retained until all data on the samples have been reported.
- 3.5.3. Chief, Organic Chemistry Section, will send notification to data requestors notifying them that analyses are complete and request that they notify ASB of the required disposition of samples. (See Form 3-4).
- 3.5.4. All samples will be disposed of in accordance with the guidelines in Section 4.

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## FORMS

### LABORATORY CHAIN-OF-CUSTODY

SECTION 3

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	OFFICIAL SAMPLE SEAL	ISIGNATURE		NOKE		3
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Form 3-1

Total
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SEPA REGION 4	
U.S. ENMRONMENTAL PROTECTION	AGENCY

#### CHAIN OF CUSTODY RECORD

ENVIRONMENTAL SERVICES DIVISION COLLEGE STATION ROAD ATHENS, GEORGIA 30613-7799

PROJECT NO. PROJECT LEADER									RE	MAR	KS				• • • • • • • • • • • • • • • • • • • •		_		_			_		
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DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory. Pink copy retained by laboratory. White copy is returned to complete; Yellow copy retained by samplers.

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#### CUSTODY ROOM SAMPLE LOG

Bottle Type/ Parameter	Out Date/Time	In Date/Time	Date Consumed	Date Disposed	Name
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#### DISPOSAL LETTER

#### UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION IV COLLEGE STATION RD. ATHENS, GA 30613

****MEMORANDUM****										
SUBJECT: Sample Disposal Request										
FROM:	Sandra	Fitzgeral	.d							
THRU:	Tom B.	Bennett,	Jr.							
TO:				,						
All requested data from the projects listed below have been reported to you. Please advise of sample disposition by checking the Disposal or Hold column to the right of the Project, then signing and returning this memo ASAP.										
Projec	t Pr	roject Name	•	City	St.	Dispose	Hold			
Disposal Authorized by: Date										

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## FIGURES

### LABORATORY CHAIN-OF-CUSTODY

SECTION 3

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COMPUTER	TAG	SCREEN

LABORATORY	SAMPLE DA	TA HANAGEHEN	r system logo	ing screen		
SAD *****	** Proje	cr #	_ PROG ELEM	ent #	CONFIDENTIAL	
STATION			·		STORET #	_
SOURCE						_
CITY		STATE	TYPE CODE	U/H	SAMPLE TYPE	_
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		END TIME				
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REMARKS					·	
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EP TOX? (Y	FOR YES)					
ANALYSIS:						
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#### 4. GENERAL LABORATORY PRACTICES

- 4.1. Intrinsic to the production of quality analytical data is the quality of laboratory services available to the analyst. Without adequate quality control being exercised with regard to facilities, services, laboratory environment, instrumentation, and laboratory supplies, an analyst cannot be expected to produce reliable analytical data.
- 4.2. Recognizing the necessity of maintaining control over general laboratory operation, the subsequent sections outline provisions for maintaining the quality laboratory support services.
- 4.3. All quality control checks listed in this section should be recorded in the Laboratory General QA Logbooks.

#### 4.4. Laboratory Apparatus and Instruments

#### 4.4.1. Incubators and Waterbaths

- 4.4.1.1. If an automatic temperature recorder is not used, place calibrated thermometer with bulb immersed in liquid on a central shelf and record temperature twice daily (a.m. and p.m.) when the incubator is in use.
- 4.4.1.2. Periodically check temperature variations when incubator or waterbath is loaded to capacity.
- 4.4.1.3. Drain and clean waterbath as required and refill with laboratory pure water.

#### 4.4.2. Refrigerators and freezers

- 4.4.2.1. Check temperature weekly.
- 4.4.2.2. Clean periodically and discard outdated materials.
- 4.4.2.3. Do not store food in any laboratory refrigerator or freezer.

#### 4.4.3. Autoclave and Hot Air Oven (Bacteriological)

- 4.4.3.1. Record date, and sterilization time, and temperature for each cycle.
- **4.4.3.2.** Operate hot air oven at a minimum of 170°C for sterilization.

#### 4.4.4. Balances

- 4.4.4.1. Check with Class-S weights at least monthly and record in the QC log.
- 4.4.4.2. Clean and level balances as required.

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4.4.4.3. Maintain annual maintenance services contract.

#### 4.4.5. pE Meters

- 4.4.5.1. Date all pH buffer solutions when opened.
- 4.4.5.2. Standardize meter with pH 7.0 and pH 4.0 and/or pH 10 buffer before each use.
- 4.4.5.3. Use pH buffer aliquot only once.

#### 4.4.6. Thermometers

4.4.6.1. Check all laboratory thermometers annually with a reference National Bureau of Standards thermometer or one which meets the requirements of NBS Monograph 150. Mark any necessary corrections on each thermometer and record in the QC logbook.

#### 4.5. Laboratory Supplies

#### 4.5.1. Glassware

- 4.5.1.1. Glassware used in general laboratory operations must be of a high quality borosilicate glass, e.g., "Pyrex" or "Kimax." Volumetric glassware must be of a Class "A" quality.
- 4.5.1.2. Clean glassware in hot water with a suitable detergent, rinse in hot water to remove detergent residue, and finally rinse in laboratory pure water. Glassware used in special analyses, e.g., metals and organics require more scrupulous cleaning., e.g., acid and/or solvent washing. Glassware must be oven-dried or drained thoroughly before use or storage.
- 4.5.1.3. Perform the Inhibitory Residue Test on initial use of a washing compound and whenever a different formulation of washing compound is used in order to insure that microbiological glassware is free of toxic residue.

### 4.5.2. Chemicals, Reagents, Solvents, Standards, Gases, and Culture Media

- 4.5.2.1. The quality of chemicals, reagents, solvents, standard gases, and culture media used in the laboratory is determined by the sensitivity and specificity of analytical technique being used. Reagents of lesser purity than specified by a method will not be used.
- 4.5.2.2. Reagents and culture media (excluding high-demand items) should be purchased in small quantities to minimize extended shelf storage.
- 4.5.2.3. Date all reagents, standards, and culture media when received and when opened or prepared, and discard when outdated, or when evidence of discoloration or deterioration is detected.

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4.5.2.4. Record lot number, date, volume, weight, sterilization time temperature, final pH, and analyst's name for each batch of bacteriological media or other materials. Perform sterility checks on each batch, and record results in the media QC log.

#### 4.5.3. Laboratory Pure Water

- 4.5.3.1. The laboratory pure water system consists of a reverse osmosis and deionization supply followed by a carbon module, two ion exchange modules and a membrane filter unit with a pore size of 0.22 um (Milli-Q). The system is also equipped with a direct reading resistivity meter.
- 4.5.3.2. Change system modules as recommended by the manufacturer or as indicated by water quality. Date modules when changed.

#### 4.6. Laboratory Hazardous Westes Handling and Disposal Procedures

- 4.6.1. It is the policy of the Analytical Support Branch to collect, store, package, label, ship and dispose of hazardous wastes in a manner which ensures compliance with all Federal, State and local laws, regulations and ordinances. These procedures are also designed to minimize employee exposure to hazards associated with laboratory generated hazardous wastes and to afford maximum environmental protection.
- 4.6.2. Policies and procedures for operation of the Division's environmental compliance program are detailed in the document, AERL/Region IV Environmental Compliance Policies and Procedures Manual, May, 1989.

#### 4.6.3. Regulatory Requirements

4.6.3.1. ASB is subject to the Resource Conservation and Recovery Act regulations as contained in the Georgia Rules for Hazardous Waste Management for the handling, storage and disposal of laboratory-related hazardous wastes. Generally, the laboratory is subject to the rules applicable to generators of 100-1000 kg/mo.

#### 4.6.4. Waste Handling Practices

- 4.6.4.1. Hazardous Waste Determination. The determination of whether or not a waste is a regulated substance is made by applying the following criteria either individually or in combination:
  - 4.6.4.1.1. Is the waste material listed in 40 CFR 261.30 261.33(e)?
  - 4.6.4.1.2. Does the material conform to any of the listing characteristics specified in 40 CFR 261.20 261.24?
  - 4.6.4.1.3. Does the generator have personal knowledge of the hazardous nature of the material?

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4.6.4.1.4. Would disposal of the material as non-regulated waste pose an environmental threat and/or leave the Agency open to criticism?

4.6.4.2. Wastes which meet any of the above criteria must be handled and disposed of as a regulated waste.

#### 4.6.5. Waste Minimization

- 4.6.5.1. The Branch Chief is responsible for ensuring adherence with all Region IV waste handling and disposal requirements for all laboratory operations. This responsibility includes the implementation of procedures (i.e., technical and/or management) designed to minimize the generation of hazardous wastes.
- 4.6.5.2. Waste minimization should be a prime consideration of initial experimental design and investigation planning. The degree to which waste minimization is achieved ultimately impacts the operational and cost effectiveness of our overall hazardous waste management program.

#### 4.6.6. Tracking

- 4.6.6.1. An automated waste accumulation tracking system (HAZWASTE) is maintained to account for monthly and annual hazardous waste generation. This information database is maintained by the Region IV Analytical Support Branch (ASB). Information maintained in the system includes: waste batch, date waste placed in storage, individual responsible for generating the waste, description (contents) of the waste, quantity of waste, EPA Hazardous Waste Number, date waste shipped, manifest numbers, and TSD Pacility receiving wastes.
- 4.6.6.2. Sam Dutton (ASB, Room 261) is responsible for initial waste logging and acceptance for storage.

#### 4.6.7. Waste Accumulation Limits

4.6.7.1. As a small quantity generator, the laboratory is subject to the following waste accumulation limits:

#### 4.6.7.1.1. Hazardous waste

- 4.6.7.1.1.1. Generate no more than 1000 kg/mo and accumulate no greater than 6000 kg of wastes. Wastes must be disposed of within 180 days of the start of accumulation, or within 270 days if waste is transported more than 200 miles for disposal.
- 4.6.7.1.1.2. Wastes generated in excess of these limits subjects the laboratory to the full generator rules (40 CFR 262.34 (a)).

#### 4.6.7.1.2. Acutely Hazardous Waste

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4.6.7.1.2.1. Those wastes specifically listed in 40 CFR 261.31 and 261.33 (e)(f) are considered acute hazardous waste. The laboratory cannot generate more than 1 kg/mo of acute hazardous waste and retain its' small quantity generator status. The 180/270-day storage limit also applies to acutely hazardous wastes if less than 1 kg/mo is generated. If more than 1 kg of acutely hazardous waste is generated during one month, these wastes must be disposed of within 90 days.

4.6.7.1.3. Waste accumulation will be monitored to ensure that the applicable generation and accumulation (i.e., quantity/time) limits are not exceeded. Waste will be disposed of as required to ensure conformance with the regulatory limits (i.e., 180 days) and at a minimum of twice per year.

#### 4.6.8. Waste Packaging and Labeling

- 4.6.8.1. All hazardous wastes designated for satellite and/or temporary storage must be packaged in an appropriate container designed to avoid loss or spillage of the materials. Each container must be labeled with the information required. If the waste contains PCB's this should be highlighted on the label and the container marked with the appropriate PCB label according to 40 CFR 761.40. Refer to, AERL/Region IV Environmental Compliance Policies and Procedures Manual, May, 1989 for specific PCB labeling and marking requirements. The label should also contain any precautionary handling or storage requirements the generator thinks are appropriate. Labels can be obtained from the Waste Control Officer.
- 4.6.8.2. Before transporting or offering a hazardous waste for off-site transport, all wastes must be packaged, labeled and marked in accordance with DOT requirements (49 CFR Parts 172, 173, 178 and 179). The Waste Control Officer will ensure that all containers shipped off-site are properly packaged and labeled and that the transport vehicle is appropriately placarded and manifest documentation is complete.

#### 4.6.9. Waste Storage

- 4.6.9.1. Except for in-laboratory accumulation (i.e., satellite storage (40 CFR 262.34 (c)(1)), all hazardous wastes generated at the Region IV, College Station Road facility and accumulated for disposal will be stored in the Hazardous Waste Storage Building (HWSB). The HWSB is located adjacent to and detached from the main AERL building. The building is specifically designed for the storage of hazardous materials.
- 4.6.9.2. Materials stored in the HWSB are segregated according to compatibility groups.
- 4.6.9.3. The hazardous waste storage facility will be inspected on a weekly basis as required 40 CFR 265 15. A record of the

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inspections will be maintained in the Hazardous Waste Storage Building Inspection Log. The Log will be maintained in the storage area. Any problems detected during the inspections will be reported in writing to the Environmental Compliance Coordinator.

- 4.6.9.4. An inspection of emergency equipment and spill control equipment will be conducted twice annually and documented in the Hazardous Waste Storage Building Inspection Log.
- 4.6.9.5. Additional housekeeping and security inspections of the HWSB will be performed on a regular basis in conjunction with safety inspections conducted by the AERL Health and Safety Committee. An inspection report will be provided to the Environmental Compliance Coordinator and the Officers in Charge.
- 4.6.9.6. Hazardous waste generated at EPA's leased facility at the U. S. Forest Service (USFS) location will be stored in the USFS Hazardous Materials Building at that site.

#### 4.6.10. Waste Disposal

- 4.6.10.1. Disposal of regulated laboratory wastes is the culmination of the waste management process. As such, selection of a responsible waste transporter and disposal facility is vitally important. The selection of a waste transporter must be predicated on their being permitted to transport hazardous wastes coupled with an absence of prior RCRA/DOT violations and a proven record of successful performance.
- 4.6.10.2. The method of waste disposal will, in part, dictate the selection of a waste disposal facility. To the extent possible, it will be the policy of Region IV to dispose of all hazardous wastes by incineration, and/or chemical treatment/fixation. Landfilling of hazardous will be avoided if at all possible. Factors considered in the selection of a waste disposal facility include: current permit status, compliance with the EPA Off-Site Policy, (SARA Sec. 121), past performance, effectiveness of treatment processes and ability to provide a certificate of disposal. To the extent possible, all hazardous waste will be disposed of at facilities which comply with the EPA off-site policy.
- 4.6.10.3. Non-regulated solid wastes will be disposed of in the building dumpster. Non-regulated aqueous wastes will be flushed to the sewer system. Spent sample containers disposed of in the dumpster should have their labels removed or obliterated.

#### 4.6.11. Recordkeeping

4.6.11.1. All records related to the generation and disposal of hazardous wastes will be retained as permanent facility records. Records to be maintained include: manifests, exception reports, waste analysis data for shipped wastes, annual reports, certificates of disposal, facility inspection/audit reports, and

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correspondence with Federal, State and Local regulatory authorities.

4.6.11.2. Records will be maintained in the files of the ASB Immediate Office.

#### 4.6.12. Contingency Measures

4.6.12.1. As required by 40 CFR 265.50 - 265.56, a Hazardous Waste Contingency Plan has been developed which outlines facility emergency response procedures.

#### 4.7. Procedures for Satellite Hazardous Waste Accumulation

- 4.7.1. Many laboratory operations necessitate the generation of hazardous wastes (e.g., solvents, acids, etc.) which are routinely accumulated near the point of generation. The in-laboratory "satellite" accumulation of such waste should be carefully controlled by the laboratory manager so as to avoid creating an unsafe situation and also comply with RCRA temporary storage requirements.
- 4.7.2. The RCRA regulations (40 CFR 262.34(c)(1)) permits the temporary accumulation of hazardous waste or acutely hazardous wastes at or near the point of generation. Waste accumulated in this manner are considered to be in "satellite accumulation."
- 4.7.3. Hazardous Wastes. The following procedures apply to satellite accumulation of hazardous waste in ASB facilities:
  - 4.7.3.1. All waste containers must be clearly marked with a red "Hazardous Waste" label. These labels are available from Sam Dutton (Room 261). The label must also indicate the date when accumulation begins.
    - 4.7.3.1.1. The contents of the container must be marked on the label. Be specific in the identification of the contents.
    - 4.7.3.1.2. All satellite storage containers must be closed except during periods of waste transfer. Some operations (e.g., AA, LC, ICP, etc.) may require using a container lid with a hole for introducing the waste via a tube. Waste collection vessels requiring zero back pressure can be fitted with an open-to-the-air absorbent trap (e.g., carbon filled).
    - 4.7.3.1.3. The volume of waste accumulated in the laboratory should not exceed 2 gallons. Exceptions would be instrument (i.e., AA, ICP) waste acid reservoirs.
    - 4.7.3.1.4. Volatile and/or flammable wastes should be temporarily stored in laboratory fume hoods nearest the point of generation.
    - 4.7.3.1.5. Caution must be exercised by the analysts to avoid creating incompatible and/or reactive waste mixtures.

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4.7.3.1.6. Waste removed from "satellite" storage for disposal will be handled according to the procedures contained in the AERL/Region IV Environmental Policies and Procedures Manual, May, 1989.

#### 4.7.4. Acutely Hazardous Wastes

- 4.7.4.1. Acutely hazardous wastes are those listed in 40 CFR 261.31-261.33 and must be accounted for separately from non-acute wastes. The following procedures apply to the satellite storage of acutely hazardous wastes:
  - 4.7.4.1.1. The acute waste must be collected in separate containers from the non-acute hazardous waste and be labeled as containing acute waste.
  - 4.7.4.1.2. Accumulation of acute waste cannot exceed one (1) quart and remain in the laboratory. Once the volume reaches one quart, the waste container must be dated and removed to the permanent hazardous waste storage area within three (3) days.
  - 4.7.4.1.3. Except for the labeling and accumulation limits, acute wastes will be handled in the same manner as hazardous wastes.
- 4.7.4.2. Laboratory managers and supervisors should conduct periodic walk-through inspections to ensure the proper application of temporary waste accumulation procedures.

#### 4.8. Guidelines for Disposal of Environmental Samples

- 4.8.1. Samples submitted to the laboratory for analysis are excluded from regulation as hazardous waste under 40 CFR 261.4(d) provided the samples are being transported to or from the laboratory, or are being analyzed, are being held for analysis, are being maintained in custody for legal reasons. However, once a decision is made to dispose of laboratory samples, the exclusion provisions of 40 CFR 261.4(d) no longer apply. Depending upon the characteristics and/or contents of such samples, they may be subject to regulation as a hazardous waste under RCRA or as a PCB-containing material under TSCA and must be handled accordingly.
- 4.8.2. Not all samples are routinely subjected to characteristic testing and are not readily classified as a hazardous waste. To address the problem of proper sample handling and disposal in the laboratory, guidelines have been developed to aid laboratory and environmental compliance personnel in making a decision whether or not to handle a particular spent laboratory sample as either a RCRA regulated or non-regulated waste containing potentially hazardous/toxic substances or simply a solid waste. Application of these guidelines provides an environmentally conservative approach to the disposal of spent laboratory samples and minimizes the potential for non-compliance with RCRA regulations.

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- 4.8.3. Once the laboratory receives approval from a project leader to dispose of samples, the laboratory generates a report from its laboratory information management system (SAAMS) which describes each analysis performed on the individual samples together with a parameter by parameter listing of positive results. Using the analysis information, a decision can be made as to how the samples must be handled for disposal.
- 4.8.4. The handling and disposal guidelines are as follows:
  - 4.8.4.1. All discarded soil, sediment or waste samples are considered hazardous wastes if they meet any of the following criteria:
    - 4.8.4.1.1. Fail any of the listing characteristic tests (i.e., corrosivity, ignitability, toxicity characteristics (TC), reactivity) described in 40 CFR 261.21 261.24, or
    - 4.8.4.1.2. Is a waste listed in 40 CFR 261.31 33.
    - 4.8.4.2. Discarded samples meeting the above criteria will be labeled, stored, manifested, transported and disposed of as RCRA-regulated hazardous wastes. To the extent possible, these materials will be disposed of by incineration.
    - 4.8.4.3. Discarded samples not failing any of the characteristic tests described in 40 CFR 261.21 261.24 or listed in 40 CFR 261.31-33 but meeting the criteria listed below will be handled as non-regulated wastes containing potentially hazardous and/or toxic substances.
      - 4.8.4.3.1. Contain cyanide at any concentration, or
      - 4.8.4.3.2. Contain pesticides or herbicides in a cumulative concentration =/> 0.5 mg/kg (ppm), or
      - 4.8.4.3.3. Contain other organic compounds (i.e., non-pesticide, herbicide) in a cumulative concentration =/> 50 mg/kg (ppm), or
      - 4.8.4.3.4. Contain or is suspected of containing dioxins, furans, or pentachlorophenol (PCP)
      - 4.8.4.3.5. Contain the following metals at or above the listed concentrations: arsenic 100 mg/kg, barium 2000 mg/kg, cadmium 20 mg/kg, chromium 100 mg/kg, Lead 100 mg/kg, mercury 4 mg/kg, selenium 20 mg/kg, and/or Silver 100 mg/kg.
      - 4.8.4.3.6. Is toxic at concentrations < 20% (EC or LC50) using aquatic and/or terrestrial bioassay techniques, or
      - 4.8.4.3.7. Is an oil.
- 4.8.5. Discarded samples meeting the criteria listed in 4.8.4.3 will be labeled and manifested as "non-regulated wastes, containing

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potentially hazardous substances"; however, they will be handled and disposed of in the same manner as a regulated hazardous waste.

- 4.8.6. Discarded solid samples not meeting the criteria listed 4.8.4.1 and 4.8.4.3 above will be disposed of as solid wastes.
- 4.8.7. All acidified water samples are disposed of via the sanitary sewer following elementary neutralization. This method of disposal is applicable to all water samples which are classified as a hazardous wastes exclusively on the basis of corrosivity. Elementary neutralization of such samples is applicable under 40 CFR 265.1(c)(10) and 260.10.
- 4.8.8. All non-acidified water samples are disposed of via the sanitary sewer.
- 4.8.9. All samples containing total PCB's greater than 50 mg/kg are subject to TSCA provisions contained in 40 CFR 761 and are disposed of as PCB containing materials.

#### 4.9. <u>Elementary Neutralization Procedures</u>

- 4.9.1. The RCRA regulations (40 CFR 265.1(c)(10)) allows for elementary neutralization of acidified wastes which are classified as hazardous wastes exclusively on the basis of corrosivity. The laboratory has several waste streams which are hazardous based on corrosivity alone and are subjected to elementary neutralization.
- 4.9.2. In the atomic absorption and induced plasma elemental analysis operations, an acidified aspiration wastewater stream is produced. These waste streams typically have a pH of less than 2. Since these waste steams are RCRA regulated only on the basis of corrosivity, elementary neutralization followed by discharge to the sewer is the most effective and reasonable approach to handling these wastes.
- 4.9.3. Elementary neutralization is conducted in the following manner:
  - 4.9.3.1. Inductively Coupled Plasma (ICP) Liquids:
    - 4.9.3.1.1. Aspiration liquid is collected in 5 gallon carboys stationed at the individual instruments. The carboys are capped except for an opening to allow for the waste to enter via a tygon tube and a small opening to allow for pressure equalization.
    - 4.9.3.1.2. Each of the collection carboys are labeled with a Hazardous Waste label.
    - 4.9.3.1.3. When filled, the carboys are transferred to a fume hood for neutralization.
    - 4.9.3.1.4. Approximately one liter of 2 N sodium hydroxide is carefully added to the waste and mixture is stirred. The pH of the mixture is checked with indicator paper and additional

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increments of 2 N sodium hydroxide added until the pH of the solution is near neutral.

- 4.9.3.1.5. The neutralized wastes are then disposed of via the laboratory sewer.
- 4.9.4. The 5 gallon polyethylene carboys are designated as elementary neutralization units and conform with the definition specified in 40 CFR 260.10..

#### 4.9.5. Atomic Absorption Pluids:

- 4.9.5.1. Aspiration liquid from atomic absorption analyses (e.g., mercury) is a mixture containing concentrated sulfuric acid. The waste is carefully poured into a large laboratory sink equipped with a tall stopper.
- 4.9.5.2. Dolomitic lime (mineral calcium magnesium carbonate) is slowly and carefully added to the acid mixture. The pH of the mixture is periodically checked with indicator paper until the pH of the solution is near neutral.
- 4.9.5.3. The neutralized waste is then disposed of via the laboratory sewer.
- 4.9.6. The laboratory sink is designated as the elementary neutralization unit and conforms with the definition specified in 40 CFR 260.10.
- 4.9.7. The same elementary neutralization procedure described in 4.9.5. above is applicable to discarded acidified water samples classified as hazardous exclusively on the basis of corrosivity.
- 4.9.8. Extreme caution must be exercised by personnel conducting the neutralization procedures to avoid skin contact with the acid mixtures.

### 4.10. <u>Handling</u>, <u>Storage</u>, <u>Disposal and Reporting Procedures for PCB</u> <u>Containing Materials</u>

4.10.1. The handling, storage, disposal and reporting of PCB items, containers, and articles containing PCB's in concentrations greater than 50 ppm are regulated under the Toxic Substances Control Act (TSCA). Applicable regulations are contained in 40 CFR Part 761. The handling of PCB materials is viewed as a sub-element of the ASB hazardous waste handling and disposal program.

#### 4.10.2. REGULATORY REQUIREMENTS

- 4.10.2.1. Marking Requirements: Any PCB article or container (40 CFR 761.3) of PCB materials in a concentration greater than 50 ppm must be properly marked according to 40 CFR 761.40.
- 4.10.2.2. Storage Requirements: Any PCB containing material

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disposed of within 1 year from the date it was first placed in storage (40 CFR 761.65 (a).

- 4.10.3. The storage facility shall comply with the requirements specified in 40 CFR 761.65 (b) (1) (e.g., roof, walls, floor, curbing, location, marking, inspection, etc.).
  - 4.10.3.1. Temporary Storage: PCB wastes stored in laboratories are considered to be in temporary storage as described in 40 CFR 761.65 (c) without having to comply with the storage requirements provided that: (1) the wastes container displays a proper PCB label, (2) contains the date accumulation started, (3) are stored in a DOT specification container as described in 40 CFR 761.65 (c)(6), and (4) are not stored in the laboratory for more than 30 days.
  - 4.10.3.2. Reporting and Records: If at any time the facility stores 45 kg (99.4 pounds) of PCB material with a concentration greater than 50 ppm, the following information will be compiled in an annual report: volume of PCB's stored, storage dates, disposal dates, and PCB source [40 CFR 761.180(a)]. An Annual Report will be prepared by July 1 each year.
  - 4.10.3.3. Disposal Requirements: Destruction of PCB containing materials must be done in an incinerator which complies with the requirements contained in 40 CFR 761.70.
  - 4.10.3.4. Spill Prevention, Control and Countermeasure Plan (SPCC): As specified in 40 CFR 761.65 (c)(7)(ii) a SPCC Plan must be developed and implemented. The AERL/Region IV SPCC Plan is contained in <u>AERL/Region IV Environmental Compliance Policies and Procedures Manual</u>, May, 1989.

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#### 5. LABORATORY EQUIPMENT MAINTENANCE AND SERVICE

5.1. Proper maintenance of laboratory instrumentation is a key ingredient to both the longevity of the instrumentation, as well as, providing the analyst with equipment capable of producing reliable analyses. Proper equipment maintenance requires an alert analytical staff which recognizes the need for equipment maintenance coupled with available support services provided either by inhouse personnel or vendor specialists.

- 5.2. Responsibility for maintenance and repair of all Branch laboratory equipment is shared by the analysts and a resident electronic technician. On occasion, vendor specialists are utilized.
- 5.3. The primary elements of the equipment maintenance program include:
  - 5.3.1. All major equipment receives a daily check for such things as: cooling fan operation, pump operation, indicator readings, mechanical checks, clean air filters, etc.
  - 5.3.2. Service schedules are established for performing routine preventative maintenance on all major equipment items.
  - 5.3.3. Records are maintained for all equipment repairs.
  - 5.3.4. Instrument utilization records; including operating, and downtime, are maintained for all GC, AA, GC/MS and ICAP instruments.
  - 5.3.5. A conservative inventory of critical spare parts is maintained for high-use instrumentation.
  - 5.3.6. Vendor operation and maintenance manuals are maintained for all laboratory instrumentation.

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#### 6. LABORATORY SAFETY

#### 6.1. INTRODUCTION

- 6.1.1. All Branch employees must accept the responsibility for acting in accordance with safety rules and practices and for reporting any observed safety hazard. This section highlights some general guidelines and rules that specifically apply to the Analytical Support Branch. Obviously no set of rules will cover all possible situations. Therefore, in addition to adhering to these rules, each person is expected to exercise good judgement in all situations and to maintain a high level of safety consciousness.
- 6.1.2. The rules and guidelines listed in this section only supplement or highlight the following official publications:
  - 6.1.2.1. <u>Safety and Health Manual, ERL, Athens, Georgia, May</u> 1984. (In revision)
  - 6.1.2.2. EPA Occupational Health and Safety Manual, October 1984.
  - 6.1.2.3. Laboratory Health Monitoring Requirements, March 6, 1987.

#### 6.2. General

- 6.2.1. Lab Coats and safety glasses should be worn at all times in laboratories. The only exception to this is when personnel are working at computer terminals or microscopes. When working with corrosives and/or toxic substances, lab coats should be left in the laboratory.
- 6.2.2. Open sandals and shorts will not be worn in laboratories.
- 6.2.3. When working in any of the laboratories, it is recommended that all jewelry be removed and that personnel wash their hands frequently. Always wash hands thoroughly when leaving the laboratory.
- 6.2.4. When working with flammable materials, nylon or other totally synthetic clothing should be avoided to minimize the possibility of static sparks.
- 6.2.5. All containers should be labeled as to contents, with particular care to note corrosive or hazardous materials.
- 6.2.6. There will be no eating, drinking, or smoking in any laboratory.
- 6.2.7. Glassware that is chipped but still usable, must be fire polished before use; otherwise it must be discarded.
- 6.2.8. Never use any lab glassware as a container for food or drink.

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- 6.2.9. An inventory of all chemicals maintained in the laboratory will be prepared and updated on an annual basis.
- 6.2.10. Return all chemicals to their proper storage areas after use.
- 6.2.11. Never pipet by mouth.
- 6.2.12. Designated personnel are to conduct a safety inspection of their laboratory at least quarterly.
- 6.2.13. No perchloric acid or perchlorate salts will be stored in Analytical Support Branch. If at any time these chemicals are required in a method, special precautions will be necessary and should be coordinated with the Chief of the Inorganic Chemistry or Organic Chemistry Section.
- 6.2.14. All work areas should be cleaned at the end of each work day. Spills should be cleaned up immediately.
- 6.2.15. Samples should be in laboratories only during preparation and analysis; otherwise, keep them in the custody room, or proper volatile organic storage area.
- 6.2.16. All stock standards of a toxic nature should be prepared in a hood and stored in designated areas. Only experienced personnel should handle these standards.
- 6.2.17. Work of a hazardous nature will not be performed in a laboratory after normal business hours when only one person is present.
- 6.2.18. New personnel must be familiarized with safety practices, location of safety equipment, and made aware of possible hazards in the areas in which they will be working.

#### 6.3. Sample Receiving and Logging

- 6.3.1. When possible, determine the source of the samples and any special hazards that might be associated with them.
- 6.3.2. Some samples, especially domestic waste when sealed in containers will build up pressure. Care should be taken in handling these type samples.
- 6.3.3. Broken samples should be handled with protective gloves and disposed of immediately according to the waste disposal procedures.
- 6.3.4. The laboratory is equipped with a partial containment lab to accommodate the extraction of hazardous waste samples. Any samples of suspected of containing high levels of hazardous contaminants should be stored in that laboratory.

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6.4.1. Compressed gases should be handled in accordance with Chapter 3 of the <u>Safety and Health Manual</u>, <u>Environmental Research Laboratory</u>, Athens, GA, May 1984.

**6.4.2.** The Chief of the Analytical Support Branch will designate three people to coordinate the routine inventory of compressed gases (Mr. Tim Slagle, Mr. Lavon Revells and Mr. Mike Wasko).

#### 6.5. Radioactivity

- 6.5.1. Electron Capture detectors require wipe tests for radioactivity every six months.
- 6.5.2. The Chief of the Organic Chemistry Section will designate one person to be responsible for the wipe tests and to maintain documentation of the tests (Mr. Art Burks).

#### 6.6. Laboratory Waste Disposal Practices

6.6.1. The Chief of the Analytical Support Branch will designate a chemist to be responsible for hazardous waste disposal (Mr. Sam Dutton). (See Section 4.)

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#### 7. METHODOLOGY

7.1. A detailed listing and discussion of specific chemical and microbiological methods are not included in this manual. Instead, lists containing the methods (and analytical technique) used in this laboratory for organic analysis of all sample types are listed in Table 7-1. Table 7-1 contains the method tracking number and method summary. The method tracking number is listed in extraction logbooks to identify the organic methods of extraction and analysis. (See Table 7-2.) Table 7-3 lists all microbiological methods. See Section 11 for references to all inorganic methods.

7.2. Details on the applications, limitations, precision, and accuracy are found within the listed methods.

#### 7.3. Reporting Units

7.3.1. Table 7-4 lists the reporting concentration units for all parameters in waters, soil/sediments, fish, and waste. Also, STORET numbers are listed. These units are always to be used unless sample matrix or methodology criteria require a change. Changes in units must be coordinated between the Organic and Inorganic Chemistry Sections.

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# TABLES

# METHODOLOGY

SECTION 7

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#### LIST OF ORGANIC TEST PROCEDURES

	=	TOT OF CHARLES	1201 1100000100	
_				Method *
Par	ameter	Method	Reference	Tracker #
	Surface	Water, Monito	ring Wells, Wastewater	:
1.	Extractable Organics	Capillary GC/	'MS 8270/625/CLP	47
2.	Volatile Organics	Capillary GC/	MS 8260/624/CLP	46A
3.	Organochlorine Pesticides/PCBs	Capillary GC/	ECD 8081/608/CLP	55
4.	Acid Herbicides	Capillary GC/	ECD 8151/615	382
5.	Organophosphorus Pesticides	Capillary GC/	NPD 8141/614	57
6.	Formaldehyde	Capillary GC/	MS ASB Method FORM-10/83	48
7.	Carbamate Pesticides	HPLC	8318 and/or 63	32 61
		<u>Drinkin</u>	<u> Water</u>	
1.	Extractable Organics	Capillary GC/	MS 525/8270	47
2.	Volatile Organics	Capillary GC/	MS 524.2/8260	46 <b>λ</b>
3.	Organochlorine Pesticides/PCBs	Capillary GC/	ECD 508/8081	55
4.	Acid Herbicides	Capillary GC/	ECD 515.1/8151	38 <b>A</b>
5.	EDB and DBCP	Capillary GC/	ECD 504	52 <b>A</b>
6.	Screening for PCBs	Capillary GC/	ECD 508A	62

^{*} See Table 7-2 for method name.

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#### LIST OF ORGANIC TEST PROCEDURES

Par	ameter	Method	Reference	Method Tracker
		SEDIMENT/SO	<u>п</u>	
1.	Extractable Organics	Capillary GC/MS	3550/8270/CLP	43,43A,54
2.	Volatile Organics	Capillary GC/MS	8260/CLP	43D,54B
3.	Organochlorine Pesticides/PCBs	Capillary GC/ECD	3550/8080/CLP	43,43A,43B 43C, 54
4.	Acid Herbicides	Capillary GC/ECD	8151	51 <b>A</b>
5.	Organophosphorus Pesticides	Capillary GC/NPD	3550/8141	57
6.	Pormaldehyde	Capillary GC/MS	ASB Method FORM-10/83	48
7.	PCBS for TSCA	Capillary GC/ECD	3540/8080	31
		WASTE		
1.	Extractable Organics	Capillary GC/MS	3580/8270 3550/8270	54 <b>A</b> 54C
2.	Volatile Organics	Capillary GC/MS	8260	54B
3.	Organochlorine Pesticides/PCBs	Capillary GC/ECD	3580/8080 3550/8080	54 <b>λ</b> 54C
4.	Acid Herbicides	Capillary GC/ECD	8151	51 <b>A</b>
5.	PCBs in Waste Oil	Capillary GC/ECD	600/4-81-045 8080	35
		TISSUE		
1.	Extractable Organics	Capillary GC/MS	ASB Sonicator 8270	44
2.	Volatile Organics	Capillary GC/MS	ASB Method/8260	44
3.	Organochlorine Pesticides/PCBs	Capillary GC/ECD	ASB Sonicator 8080	44

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#### LIST OF ORGANIC TEST PROCEDURES

Par	ameter	Method	Reference	Method Tracker #
		AIR		
1.	Extractable Organics	Capillary GC/MS	PUF by TO13 8270	50C
2.	Volatile Organics	Capillary GC/MS	Canister by TO14/8260	56
3.	Organochlorine Pesticides/PCBs	Capillary GC/ECD	FUF by TO4 8080	50
4.	Formaldehyde	HPLC/UV	Trap by TO11	59

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#### ORGANIC METHOD REFERENCES, REGION IV METHOD TRACKER

#### METHOD NO.

#### HETHOD NAME

- 07 LSB-WSS, 12/74 DISTILLATION METHOD FOR WATER SOLUBLES IN WATER.
- 16 FDA-212.13.A EXTRACTION OF PESTICIDE RESIDUES FROM HIGH-MOISTURE PRODUCTS.
- 18 LSB-DAI, 1/72 DIRECT AQUEOUS INJECTION OF WATER SAMPLES.
- 31 EPA-SPCB-2/78 SOXHLET EXTRACTION FOR PCB'S IN SPILLED MATERIAL, ANALYSIS BY METHOD 55.
- 31A EPA-8080/3540-9/86 SOXHLET EXTRACTION (1:1 ACETONE/HEXANE) FOR PCBS.
- 31B EPA-8080/3541, SOXTEC EXTRACTION FOR PCBS.
- 31C EPA-8270/3540-9/86 SOXHLET EXTRACTION (METHYLENE CHLORIDE) FOR SEMIVOLATILES
- 31D SPA-8080/3540-9/86 SOXHLET EXTRACTION (HEXAME) OF FISH FOR OC PEST/PCBS.
- 35 EPA-PCB-2/81 ANALYSIS OF PCB'S IN TRANSFORMER FLUIDS & WASTE OIL.
- 38 EPA-615-1/82 EXTRACTION AND ANALYSIS OF CHLORINATED HERBICIDES IN WATER (ASB MODIFICATIONS).
- 38A EPA-8151-90 MODIFICATION OF HERBICIDE METHOD FOR HYDROLYSIS & W/CAPILLARY COLUMNS.
- 43 EPA-3550-10/89 LOW CONC. SONICATOR EXTRACTION OF SED./SOIL, ANALYSIS BY 55 & 47.
- 43A LOW CONC. SONICATOR EXTRACTION OF SED./SOIL WITHOUT GPC, ANALYSIS BY  $55 \pm 47$ .
- 43B LOW CONC. SONICATOR EXTRACTION OF SED./SOIL WITH SULFURIC ACID CLEANUP, ANALYSIS BY 55.
- 43C LOW CONC. SONICATOR EXTRACTION OF SED./SOIL USING HEXANE/ACETONE, ANALYSIS BY 55.
- 43D LOW CONC. PURGE & TRAP ANALYSIS OF SED./SOIL. ANALYSIS BY 46A.
- 44 ASB-0B-1/90 SONICATOR EXTRACTION OF TISSUE, ANALYSIS BY 55, 46A, & 47.
- 44A SONICATOR EXTRACTION OF TISSUE USING HEXAME, ANALYSIS BY 55.
- 44B SONICATOR EXTRACTION OF TISSUE USING HEXANE WITH SULFURIC ACID CLEANUP, ANALYSIS BY 55.
- 45 EPA-617-1/82 EXTRACTION & GC/EC ANALYSIS OF CHLOR. PEST./PCBS IN WATER. (ASB MODS.)
- 46 EPA-624-7/82 PURGE & TRAP GC/MS ANALYSIS OF WATER BY PACKED COLUMN.
  (ASB MODS.)
- 46A EPA-8260-12/87 PURGE & TRAP GC/MS ANALYSIS OF WATER BY CAPILLARY COLUMN.
- 46B EPA-8260/8030 HEATED P & T GC/MS ANALYSIS OF WATER BY CAPILLARY COLUMN.
- 47 EPA-8270-12/87 SEMIVOLATILE GC/MS ANALYSIS OF WATER BY CAPILLARY COLUMN.
- ASB-FORM-10/83 EXTRACTION AND ANALYSIS OF FORMALDEHYDE IN WATER AND SEDIM/SOIL, ANALYSIS BY METH. 47
- 48A EPA-8315-1/90 EXTRACTION AND ANALYSIS OF FORMALDEHYDE IN WATER AND SOLIDS BY HPLC.

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#### METHOD REFERENCES, REGION IV METHOD TRACKER (Continued)

#### METHOD NO.

#### METHOD NAME

- 50 EPA-TO4-4/84 EXTRACTION OF AIR PUF SAMPLES FOR CHLOR. PEST/PCBS, ANALYSIS BY 55.
- 50A EXTRACTION OF AIR PUFS WITH ETHYL ACETATE/HEXANE, ANALYSIS BY 55 AND/OR 575LA EXTRACTION OF ACID HERBICIDES FROM SED./SOIL, ANALYSIS BY 38A
- 52 EPA-501.2-11/79 TRIHALOMETHANES BY MICROEXTRACTION & GC/EC ANALYSIS.
- 52A EPA-504-/89 EDB & DBCP BY MICROEXTRACTION & GC/EC ANALYSIS.
- 52B EPA-505-/89 CHOR. PEST./PCBS BY MICROEXTRACTION & GC/EC ANALYSIS.
- 53 EPA-9020-9/86 TOX ANALYSIS.
- 54 EPA-3550-10/89 MED. CONC. SONICATOR EXTRACTION OF SED./SOIL, ANALYSIS BY 55 & 47.
- 54A EPA-3580-9/86 DILUTION OF WASTE WHEN SOLVENT SOLUBLE (NO SONICATOR).
  ANALYSIS BY 55 & 47.
- 54B EPA-8260-12/87 PURGE & TRAP GC/MS ANALYSIS OF WASTE & MEDIUM SED./SOIL.
- 54C EPA-3550-10/89 MED. CONC. SONICATOR EXTRACTION OF WASTE, ANALYSIS BY 55 AND 47.
- 55 EPA-608-10/84 EXTRACTION & GC/EC ANALYSIS OF CHLOR. PEST./PCBS IN WATER. (ASB MODS.)
- 56 EPA-TO14-5/88 ANALYSIS OF AIR CANISTER SAMPLES BY GC/MS FOR VOC (ASB MODS.) BY 46A.
- 57 EPA-507-/89 EXTRACTION & GC/NP ANALYSIS OF N & P PEST. IN WATER. (ASB MODS.)
- 58 EPA-525-7/88 DETN. OF ORGANICS IN DW BY SPE AND CAPILLARY COLUMN GC/MS.
- 59 EPA-TO11, 5/88, FOR DTERM. OF FORMALDEHYDE IN AIR USING DNPH CARTRIDGES AND HPLC.
- 60 EPA-1311 TCLP EXTRACTION, ANALYSIS BY METHOD 38A, 46A, 47, AND/OR
- 61 EPA-8318-/90 EXTRACTION AND ANALYSIS (WATER & SOIL) FOR CARBAMATE PEST. BY HPLC/UV (632).
- 62 EPA-508A-/89 SCREENING FOR PCBS BY PERCHLORINATION AND GC/ECD.

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#### METHOD SOURCES FOR TABLES 7-1 & 7-2

- 1. 1000-8000 Methods: USEPA, <u>Test Methods for Evaluating Solid Waste</u>, SW-846, 3rd Edition, 1986 plus the 1st and 2nd Updates.
- 2. 500 Methods: USEPA, Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Dec., 1988.
- 3. 600 Methods: USEPA, <u>Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act-40CFR Part 136</u>, Federal Register of Oct. 26, 1984.
- 4. TO Methods: USEPA, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-84-041, Apr. 1984 plus the Supplements of 1986 and 1988.
- 5. CLP Methods: <u>USEPA Contract Laboratory Program Statement of Work for Organics Analysis</u>, <u>Multi-Media</u>, <u>Multi-Concentration</u>, 1990.
- 6. USFDA Methods: Pesticide Analytical Manual, Volumes I and II.
- 7. Region 4 Methods: Adaptations of Published Methods when Official Methods are not Available.

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#### List of Microbiological Test Procedures

	<u>Parameter</u>	<u>Method</u> <u>F</u>	Reference	
		<u>Wastewater</u> St	d. Meth. 1/	EPA ^{2/}
1.	Pecal Coliform	MPN, 5 tube, 3 dilution Membrane Filter (MF)	908C 909C	pg. 132 pg. 124
2.	Pecal Coliform in presence of chlorine	MPN, 5 tube, 3 dilution Membrane filter (MP)	908C 909C	pg. 132 pg. 124
3.	Total coliform	MPN, 5 tube, 3 dilution Membrane filter (MF)	908A 909A	pg. 114 pg. 108
4.	Total coliform in presence of chlorine	MPH, 5 tube, 3 dilution Membrane filter (MF)	908A 909A	pg. 114 pg. 108
5.	Fecal Streptococci	MPN, 5 tube, 3 dilution Membrane filter (MF) 1/2 Plate count	910A 910B 910C	pg. 139 pg. 136 pg. 143
		Drinking Water 5/ St	d. Meth. 6/	EPA ^{2/}
1.	Total Coliform	MPN, 5 tube, 10 ml or 100 ml sample volume/tub	908A	pg. 114
		100 ml sample volume/tub Membrane filter (MF) 3/2 Minimal medium ONPG-MUG ⁷ (MMO-MUG), MPN, 5 tube, 10 ml tube	909A	pg. 108
2.	Standard Plate Count 8/	Pour Plate, 1.0 ml and 0.1 ml sample volumes	907	pg. 101

- 1/ Standard Methods for the Examination of water and wastewater, 15th Ed., 1980.
- 2/ Microbiological Methods for Monitoring the Environment, EPA-600/8-78-017.
- 3/ 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth and development.
- 4/ Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.
- 5/ On 12/31/90 there will be method additions and deletions to conform with requirements of the revised drinking water total coliform rule.
- 6/ Standard Methods for the Examination of Water and Wastewater, 14th Ed., 1975.
- 7/ Federal Register/Vol. 54, No. 135/Monday, July 17, 1989, pgs. 29998-30.
- 8/ This procedure provides a standardized means of determining the density of aerobic and facultative anaerobic heterotrophic bacteria in water.

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TEST			STORET	NOS.			REPORTI	NG UNIT	_ s
NO.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
1001	DISTRIBUTION CORFFICIENT								
1002	DENSITY (20 DEG. C)				71820	GH/HL			GH/HL
1005	ASBESTOS (FIBROUS)	81855	34228	34229		F/L	UG/KG	HG/KG	HG\KG
1006	ASH					•		•	
1007	ASBESTOS, BULKED							•	
1008	ames test								
1010	BAC-T								
1011	ODOR (60 DEGREE C)	00086				TOD			
1012	ODOR (ROOM TEMP)	00085				TOD			
1013	PH (LABORATORY)	00403				PHUN			
1014	COLOR (TRUE-PTCO)	00080				PTCO			
1015	COLOR (APPARENT-PTCO)	00081				PTCO			
1016	COLOR(ADMI @ PH 7.6)	00083				ADHI			
1017	TOXIC EQUIVALENT VALUE					PPQ	PPT	PPT	•
1018	COLOR(ADMI @ ORIG. SMPL. PH)								
1020	CONDUCTIVITY	00095				UMHOS			
1025	CORROSIVITY								PH
1030	FLASH POINT				74030				DEG C
1031	HEAT CONTENT (HEAT OF COMBUSTION)						BTU/#		BTU/ <b>₽</b>
1035	HARDNESS (AS CACO3)	00900				MG/L			
1040	SETTLEABLE SOLIDS	50086				ML/L			
1045	TOTAL SOLIDS	00500				MG/L	MG/KG		HG/KG
1046	SOLIDS								
1047	TOTAL SUSPENDED PARTICULATE								
1050	VOLATILE TOTAL SOLIDS	00505				MG/L	MG/KG		HG/KG
1055	TOTAL SUSPENDED SOLIDS	00530				MG/L	MG/KG		MG/KG
1060	VOLATILE TOTAL SUSPENDED SOLIDS	00535				MG/L	MG/KG		MG/KG
1065	TOTAL DISSOLVED SOLIDS (105 DEGREE C)	00515				MG/L	MG/KG		MG/KG
1066	TOTAL DISSOLVED SOLIDS (180 DEGREE C)	70300				MG/L			
1070	VOLATILE TOTAL DISSOLVED SOLIDS	00520				MG/L	HG/KG		HG/KG
1074	TCLP								
1075	TOXICITY(EP)	00187				MG/L			
1076	CATION EXCHANGE CAPACITY (CEC)					UG/L	HQ/KG	HG/KG	HG/KG
1080	TURBIDITY	00076				NTU			
									=

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TEST			STORET NOS.					REPORTING UNITS			
10.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE		
1085	FECAL COLIFORM, MPN/100ML	31615									
1090	FECAL COLIFORM, HF/100HL	31616									
1095	TOTAL COLIFORM, MPN/100ML	31505									
1100	TOTAL COLIFORM, MF/100ML	31501									
1105	STANDARD PLATE COUNT, 35C, 48HR/ML	31748									
1110	RADIUM-226, TOTAL	09501	09507			PC/L	PC/G				
1111	RADIUM-228, TOTAL	11501				PC/L					
1112	RADIUM-226, DISS	09503				PC/L	PC/G				
1113	RADIUM-228, DISS	B1366				PC/L	PC/G				
1115	GROSS ALPHA, TOTAL	01501	01507			PC/L	PC/G				
1120	GROSS BETA, TOTAL	03501	03507			PC/L	PC/G				
000	METALS SCAN						•				
2005	SILVER	01077	01078	34474	79704	ng/r	MG/KG	HG/KG	MG/KG		
2010	ARSENIC	01002	01003	01004	79548	UG/L	MG/KG	MG/KG	MG/KG		
2015	BORON	01022	01023	81657		UG/L	MG/KG	MG/KG	HG/KG		
2020	BARIUM	01007	01008	81658	79550	UG/L	MG/KG	MG/KG	HG/KG		
2025	BERYLLIUM	01012	01013	34252	79556	UG/L	MG/KG	MG/KG	HG/KG		
2030	CADMIUM	01027	01028	71940	79580	UG/L	MG/KG	MG/KG	HG/KG		
035	COBALT	01037	01038	81659	79593	UG/L	MG/KG	MG/KG	MG/KG		
2040	CHRONIUM	01034	01029	71939	79591	UG/L	MG/KG	MG/KG	HG/KG		
2045	COPPER	01042	01043	71937	79594	UG/L	MG/KG	MG/KG	HG/KG		
2050	HOLYBDENUH	01062	01063	<b>B1662</b>	79667	UG/L	MG/KG	MG/KG	HG/KG		
2055	NICKEL	01067	01068	01069	79671	UG/L	MG/KG	MG/KG	MG/KG		
060	LEAD	01051	01052	71936	79649	UG/L	MG/KG	MG/KG	HG/KG		
065	ANTIMONY	01097	01098	01099	79547	UG/L	HG/KG	MG/KG	HG/KG		
2070	SELENIUM	01147	0114B	01149	79703	UG/L	MG/KG	MG/KG	HG/KG		
:075	TIN	01102	01103	81663	79713	UG/L	MG/KG	MG/KG	MG/KG		
:080	STRONTIUM	01082	01083	81950	79706	UG/L	MG/KG	MG/KG	MG/KG		
:085	TELLURIUM	01064	45513	79021	79708	UG/L	MG/KG	HG/KG	HG/KG		
090	TITANIUM	01152	01153	81664	79714	UG/L	MG/KG	HG/KG	HG/KG		
095	THALLIUM	01059	34480	01073	79712	UG/L	HG/KG	MG/KG	HG/KG		
100	VANADIUM	01087	01088	<b>B1665</b>	79722	UG/L	MG/KG	MG/KG	HG/KG		
105	YTTRIUM	45515	45514	79024	79725	UG/L	MG/KG	MG/KG	HG/KG		
110	ZINC	01092	01093	71938	79726	UG/L	HG/KG	MG/KG	HG/KG		
115	ZIRCONIUM	01162	01163		79727	UG/L	MG/KG	MG/KG	MG/KG		
I					-	•	•	•			

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<u>test</u>			STORET	NOS.			PPP∩PT1	NG UNIT	== • Q
NO.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
					<u> </u>	3333334			
2120	MERCURY	71900	71921	71930		UG/L	MG/KG	MG/KG	MG/KG
2125	ALUMINUM	01105	01108	81666	79545	UG/L	MG/KG	MG/KG	MG/KG
2130	Manganese	01055	01053	81741	79651	UG/L	MG/KG	MG/KG	MG/KG
2135	CALCIUM	00916	00917	<b>B1655</b>	79581	MG/L	MG/KG	MG/KG	MG/KG
2136	CALCIUM	00916	00917	81655	79581	UG/L	MG/KG	MG/KG	MG/KG
2140	Magnesium	00927	00924	<b>B1656</b>	79650	MG/L	MG/KG	MG/KG	MG/KG
2141	Magnesium	00927	00924	81656	79650	NG/L	MG/KG	MG/KG	MG/KG
2145	IRON	74010	01170	81660	79645	MG/L	MG/KG	HG/KG	HG/KG
2146	IRON	74010	01170	81660	79645	UG/L	MG/KG	MG/KG	MG/KG
2150	SODIUM	00929	00934		79705	MG/L	MG/KG	MG/KG	MG/KG
2151	BODIUM	00929	00934		<b>79</b> 705	UG/L	MG/KG	MG/KG	MG/KG
2155	CHRONIUM, HEXAVALENT	01032	29405	78220		ng/r	MG/KG	MG/KG	MG/KG
2160	POTASSIUM	00937	00938			MG/L	HG/KG	MG/KG	MG/KG
2161	POTABSIUM	00937	00938			UG/L	MG/KG	HG/KG	MG/KG
2705	BISHUTH	01017	01014			ng\r	MG/KG	MG/KG	MG/KG
2715	CBRIUN	01112	30246			NG/L	MG/KG	MG/KG	MG/KG
2720	LANTHANUH	01182	01179			ng/r	MG/KG	MG/KG	MG/KG
2725	URANIUH, TOTAL HETAL	22706				UG/L	MG/KG		
2726	LITHIUM	01132	01133			UG/L	MG/KG		
3005	ACIDITY	70508				MG/L	HG/KG	MG/KG	HG/KG
	ALKALINITY, BICARBONATE (AS CACO3)	00425				MG/L			
3009	ALKALINITY, CARBONATE (AS CACO3)	00430				HG/L			
3010	ALKALINITY, TOTAL (AS CACO3)	00410				HG/L	MG/KG	MG/KG	MG/KG
3011	VOID RECORD - CALL DAYLOR	00610				HG/L	HG/KG	MG/KG	MG/KG
3012	BICARBONATE (AS HCO3 ION)	00440				HG/L	MG/KG		
3013	CARBONATE (AS CO3 ION)	00445				MG/L	MG/KG		
3015	AMMONIA	00610				MG/L	MG/KG	MG/KG	MG/KG
3016	AMMONIA, DISSOLVED	00608				MG/L			
3017	BROMIDE	71870				MG/L			
3018	AMMONIA, UNIONIZED(AS NH3)	00619				MG/L	va /va		V0 / V0
3020 3025	CHLORIDE	00940	00701	24226	20505	MG/L	MG/KG	NO / NO	MG/KG
3025	CYANIDE	00720 00720	00721	34326 34326	79595 79595	MG/L	MG/KG	MG/KG	MG/KG
3025	CYANIDE AVENUE TO CHIORINATION	00720	00721	34320	17075	UG/L MG/L	HG/KG	MG/KG	MG/KG MG/KG
3027	CYANIDE AMENABLE TO CHLORINATION	81208				MG/L MG/L	MG/KG	MG/KG MG/KG	MG/KG
3020	CYANIDE, FREE	61208				NG/ L	UG/KG	HG/ VA	HO) NO

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BST			STORET	NOS.			REPORTI	NG UNIT	s
10.	TEST NAME	<u>water</u>	SED	<u>FISH</u>	WASTE	WATER	SED	<u>FISH</u>	WASTE
030	FLUORIDE	00951	00949	79031		MG/L	MG/KG	HG/KG	MG/KG
033	nitrate-nitrogen	00620	00621			MG/L	MG/KG	HG/KG	HG/KG
034	NITRITE-NITROGEN	00615	00616			MG/L	HG/KG		
035	NITRATE-NITRITE NITROGEN	00630				MG/L	MG/KG	MG/KG	MG/KG
036	NITRATE-NITRITE NITROGEN, DISSOLVED	00631				MG/L			
040	ORTHO-PHOSPHATE PHOSPHORUS	00660				MG/L	MG/KG	MG/KG	MG/KG
045	SILICON(SI)	01142	01144			UG/L	MG/KG	MG/KG	HG/KG
046	SILICA(SIO2)	00956				UG/L	HG/KG	MG/KG	MG/KG
050	SULFATE	00945				MG/L	MG/KG		HG/KG
054	REACTIVE SULFIDES (AS H2S)					HG/L	MG/KG		MG/KG
055	SULFIDES	00745	00747			MG/L	MG/KG		MG/KG
056	SULPITE	00740				MG/L			
060	TOTAL DISSOLVED PHOSPHORUS	D0666				MG/L	MG/KG		MG/KG
065	TOTAL KJELDAHL NITROGEN	00625	00626			MG/L	MG/KG	MG/KG	MG/KG
066	TOTAL KJELDAHL NITROGEN, DISSOLVED	00623				MG/L			
070	TOTAL-PHOSPHORUS	00665	30291			MG/L	MG/KG	HG/KG	MG/KG
1005	BIO-CHEMICAL OXYGEN DEMAND, 5 DAY	00310	00298			MG/L	hg/kg		MG/KG
1006	BIO-CHEMICAL OXYGEN DEMAND, 5 DAY, DISS	00311				Mg/L	MG/KG		MG/KG
007	BIO-CHEMICAL OXYGEN DEMAND, 20 DAY	00324				MG/L	HG/KG		HG/KG
800	BIO-CHEMICAL OXYGEN DEMAND, 60 DAY	<b>B1276</b>				MG/L	MG/KG		MG/KG
009	BOD, CARBONACEOUS, 5 DAY	80082				MG/L			
010	CHEMICAL OXYGEN DEHAND	00340				MG/L	HG/KG		HG/KG
011	CHEMICAL OXYGEN DEHAND, DISSOLVED	00341				MG/L			
015	LINEAR ALKYL SULFONATE	38260				MG/L	MG/KG	MG/KG	MG/KG
020	OIL AND GREASE	00556	00557			MG/L	HG/KG	MG/KG	HG/KG
025	OIL IDENTIFICATION								
030	PHENOLS (4AAP)	32730	32731	32734		UG/L	MG/KG	MG/KG	HG/KG
035	TOTAL ORGANIC CARBON	00680	30243			MG/L	MG/KG	MG/KG	MG/KG
036	TOTAL ORGANIC CARBON, DISSOLVED	00681				MG/L			
037	PURGBABLE ORGANIC CARBON	B12 <b>7</b> 7				MG/L			
038	PARTICULATE ORGANIC CARBON	B0102				HG/L	MG/L		
040	TANNIN AND LIGHIN	32240				HG/L			
045	LIPIDS	45579						•	
050	TOTAL ORGANIC HALOGEN					UG/L	UG/KG		HG/KG
051						UG/L			
045 050	♦ LIPIDS					UG/L	UG/KG	•	HG/I

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<u>test</u>			STORET	NOS.			REPORTI	NG UNIT	<u>s</u> _
NO.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
4055	ACETATE					HG/L	MG/KG	MG/KG	MG/KG
4060	FDCC BLUE DYE					MG/L	MG/KG		
5000	PESTICIDES SCAN								
5001	PCB SCAN								
5005	ALDRIN	39330	39333	34680	79542	UG/L	UG/KG	MG/KG	HG/KG
5010	HEPTACHLOR	39410	39413	34687	79628	UG/L	ng\ka	HG/KG	MG/KG
5015	HEPTACHLOR EPOXIDE	39420	39423	34686	79629	ng/r	ng/kg	MG/KG	HG/KG
5020	ALPHA-BHC	39337	39076	39074	79543	UG/L	UG/KG	MG/KG	HO/KO
5025	BETA-BHC	39338	34257	34258	79557	UG/L	UG/KG	MG/KG	MG/KG
5030	GAMMA-BHC (LINDAME)	39340	39343	39785	79627	UG/L	UG/KG	HG/KG	HG/KG
5035	DELTA-BHC	34259	34262	34263	79598	UG/L	UG/KG	MG/KG	HG/KG
5040	ENDOSULFAN I (ALPHA)	34361	34364	34365	79617	UG/L	ng\kg	MG/KG	HG/KG
5045	DIELDRIN	39380	39383	39404	79607	ng/r	UG/KG	MG/KG	HG/KG
5050	4,4'-DDT (P,P'-DDT)	39300	39301	39302	79681	UG/L	ng/kg	HG/KG	HG/KG
5055	4,4'-DDE (P,P'-DDE)	39320	39321	39322	79680	UG/L	UG/KG	HG/KG	HG/KG
5060	4,4'-DDD (P,P'-DDD)	39310	39311	39312	79679	UG/L	UG/KG	HG/KG	MG/KG
5065	ENDRIN	39390	39393	34685	79619	UG/L	UG/KG	MG/KG	HG/RG
5070	ENDOSULFAN II (BETA)	34356	34359	34360	79618	UG/L	UG/KG	MG/KG	MG/KG
5075	ENDOSULFAN SULFATE	34351	34354	34355	79616	UG/L	UG/KG	MG/KG	MG/KG
5080	CHLORDANE (TECH. MIXTURE) /1	39350	39351	34682	79583	UG/L	ng/kg	MG/KG	HG/KG
5085	PCB-1242 (AROCLOR 1242)	39496	39499	34689	79686	UG/L	UG/KG	HG/KG	MG/KG
1090	PCB-1254 (AROCLOR 1254)	39504	39507	34690	79688	UG/L	UG/KG	HG/KG	MG/KG
6095	PCB-1221 (AROCLOR 1221)	39488	39491	34664	79684	UG/L	UG/KG	MG/KG	HG/KG
100	PCB-1232 (AROCLOR 1232)	39492	39495	34667	79685	UG/L	UG/KG	MG/KG	MG/KG
105	PCB-1248 (AROCLOR 1248)	39500	39503	34669	79687	UG/L	UG/KG	MG/KG	MG/KG
110	PCB-1260 (AROCLOR 1260)	39508	39511	34670	79689	UG/L	UG/KG	MG/KG	MG/KG
115	PCB-1016 (AROCLOR 1016)	34671	39514	34674	79683	UG/L	UG/KG	MG/KG	MG/KG
120	TOXAPHENE	39400	39403	34691	79717	UG/L	UG/KG	MG/KG	HG/KG
125	ENDRIN ALDEHYDE	34366	34369	34370	79620	UG/L	UG/KG	MG/KG	HG/KG
135	CHLORDENE /2	77884	81765	79006		UG/L	UG/KG	MG/KG	HG/KG
140	ALPHA-CHLORDENE /2	77951	78545	78457		UG/L	UG/KG	MG/KG	HG/KG
142	BETA CHLORDENE /2			78458		UG/L	UG/KG	HG/KG	MG/KG
145	GAMMA-CHLORDENE /2	77952	78546	78459		UG/L	UG/KG	MG/KG	HG/KG
150	1-HYDROXYCHLORDENE /2		81763	79001	79518	UG/L	UG/KG	MG/KG	HG/KG
155	GAMMA-CHLORDANE /2	39810	39811	79005	79585	UG/L	UG/KG	MG/KG	MG/KG
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<u>),</u>	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE		
60	TRANS-NONACHLOR /2	39071	39073	39072		UG/L	UG/KG	MG/KG	HG/KG		
165	ALPHA-CHLORDANE /2	39348		79025	79584	UG/L	UG/KG	MG/KG	MG/KG		
70	CIB-NONACHLOR /2	39068	39070	39069		UG/L	UG/KG	HG/KG	HG/KG		
172	OXYCHLORDANE (OCTACHLOREPOXIDE) /2	79755	78871	82029		UG/L	ng/kg	ng/kg	HG/KG		
75	METHOXYCHLOR	39480	39481	81644	7 <del>9</del> 730	UG/L	UG/KG	HG/KG	MG/KG		
.85	HEXACHLORONORBORNADIENE (HCNBD)	78127		79030	79637	UG/L	UG/KG	MG/KG	HG/KG		
90	HEPTACHLORONORBORNENE (HCNB)	78020	78602		79630	UG/L	UG/KG	Mg/kg	HG/KG		
95	OCTACHLOROCYCLOPENTENE (OCCP)	39130	78660	79012	79674	UG/L	UG/KG	MG/KG	HG/KG		
200	HEXACHLOROBENZENE (HCB)	39700	39701	34688	79633	UG/L	UG/KG	MG/KG	MG/KG		
205	2,4-D	39730	39731	39734	79731	UG/L	UG/KG	UG/KG	HG/KG		
210	SILVEX (2,4,5-TP)	39760	39761	39764		UG/L	UG/KG	MG/KG	MG/KG		
215	2,4,5-T	39740	39741			UG/L	UG/KG	HG/KG	MG/KG		
220	ENDRIN KETONE	78008	82557	78211	79739	UG/L	UG/KG	MG/KG	MG/KG		
81	AZODRIN	82186				VG/L	ug/kg	hg/kg	MG/KG		
82	CAPTAN	39640				UG/L	ng/kg	MG/KG	HG/KG		
183	DALAPON	30200	38435	38436		UG/L	UG/KG	MG/KG	MG/KG		
84	PICLORAH	39720	38930	82638		UG/L	ng/kg	Mg/kg	MG/KG		
801	BUTYLATE	81410	81411			UG/L	UG/KG	hg/kg	HG/KG		
302	HOLINATE					UG/L	UG/KG	MG/KG	HG/KG		
303	CYCLOATE	30254	81891			UG/L	ug/kg	HG/KG	HG/KG		
105	ORTHENE (ACEPHATE)	81515				UG/L	UG/KG	MG/KG	HG/KG		
310	DIQUAT	78885				UG/L	UG/KG	MG/KG	HG/KG		
311	EPTC(BPTAM)	81894	81893			UG/L	UG/KG	MG/KG	MG/KG		
312	FONOFOS		82408	82407		UG/L	UG/KG	MG/KG	HG/KG		
313	PEBULATE	79192				UG/L	UG/KG	MG/KG	HG/KG		
314	VERNAM (VERNOLATE)	30324				UG/L	UG/KG	HG/KG	HG/KG		
115	PARAQUAT	82416	82415	82414		UG/L	UG/KG	MG/KG	HG/KG		
16	PHOSDRIN	39610	82643			UG/L	UG/KG	MG/KG	HG/KG		
117	DIBROM					UG/L	UG/KG	MG/KG	HG/KG		
18	DIMETHOATE	46314	78449	38461		UG/L	UG/KG	MG/KG	MG/KG		
319	DEHETON, O, S	39560	82400	82401		UG/L	UG/KG	HG/KG	MG/KG		
122	DELNAV (DIOXATHION)	38784	38785	38786		UG/L	UG/KG	HG/KG	HG/KG		
23	TRI-ALLATE					UG/L	UG/KG	MG/KG	MG/KG		

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TEST			STORET	NOS.			<u>REPORT I</u>	NG UNIT	s
NO.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
5004		22072							
5824	CHLORDIMEFORM	77953				UG/L	UG/KG	HG/KG	HG/KG
5826	ROZOL	20055	20046	00406		UG/L	UG/KG	HG/KG	MG/KG
5827	SIHAZINE	39055	39046	82406		UG/L	UG/KG	HG/KG	HG/KG
5828	DIURON	39650	2000			UG/L	UG/KG	HG/KG	MG/KG
5829	PROMETRYNE	39057	78688			UG/L	UG/KG	HG/KG	MG/KG
5831	DISULFOTON	81888	81887			UG/L	UG/KG	HG/KG	HG/KG
5832	SULFOTEPP	82201				UG/L	UG/KG	HG/KG	HG/KG
5833	DIALLATE					ng/r	UG/KG	MG/KG	HG/KG
5836	DICAMBA	38442	38444	38445		ng/r	UG/KG	HG/KG	HG/KG
5837	HIREX	39755	39758	81645		UG/L	UG/KG	HG/KG	MG/KG
5839	DECACHLOROBIPHENYL (DCB)	79750	75054	39409		NG/L	UG/KG	MG/KG	HG/KG
5840	ISODRIN	39430	39433	81654		UG/L	UG/KG	HG/KG	MG/KG
5841	FENSULFOTHION	38797	38799	38800		ng/r	UG/KG	MG/KG	HG/KG
5842	DISULFOTHION (DISULFTON)					UG/L	UG/KG	HG/KG	MG/KG
5843	ETHOPROP	81758	82288			UG/L	UG/KG	HG/KG	MG/KG
5850	OXYCHLORDANE	79755	78871	B2029		UG/L	ng\kg	HG/KG	HG/KG
5855	OCTACHLORONAPHTHALENE		78661			UG/L	UG/KG	HG/KG	HG/KG
5856	2,4'-DDT (O,P'-DDT)	39305	39306			UG/L	ng/kg	HG/KG	HG/KG
5857	2,4'-DDE (O,P'-DDE)	39327	39328	81760		ng/t	ng/kg	MG/KG	MG/KG
5858	2,4'-DDD (O,P'-DDD)	39315	39316	39325		UG/L	UG/KG	MG/KG	HG/KG
5859	TOTAL DDT RESIDUES (TDDTR)					UG/L	ug/kg	MG/KG	HG/KG
5860	DIAZINON	<b>3957</b> 0	39571	81806		UG/L	UG/KG	HG/KG	HG/KG
5861	ATRASINE	39033	39631	82404	72549	UG/L	UG/KG	MG/KG	HG/KG
5862	BROMACIL	30234				UG/L	UG/KG	MG/KG	hg/kg
5863	CYANAZINE	81757				UG/L	UG/KG	HG/KG	HG/KG
5864	PROPABINE	38535	82534	82533		UG/L	UG/KG	HG/KG	HG/KG
5865	METHYL PARATHION	39600	39601	81809		UG/L	UG/KG	HG/KG	MG/KG
5866	ORDRAM	82199				UG/L	UG/KG	HG/KG	MG/KG
5867	ALACHLOR (LASSO)	77825	<b>B1407</b>	82571		UG/L	UG/KG	HG/KG	HG/KG
5868	BENOMYL (BENLATE)	38706	38708	38709		UG/L	UG/KG	MG/KG	HG/KG
5869	CHLOROTHALONIL					UG/L	UG/KG	HG/KG	HG/KG
5870	PARATHION (ETHYL)	39540	39541	81810		UG/L	UG/KG	HG/KG	HG/KG
5871	CARBOPHENOTHION (TRITHION)	39786	39787	81898		UG/L	UG/KG	HG/KG	HG/KG
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EST			REPORTING UNITS						
<u>o.</u>	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
872	ETHION	39398	39399	46335		UG/L	ug/kg	MG/KG	HG/KG
873	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	38437	38440	38441	:	UG/L	UG/KG	HG/KG	MG/KG
874	CHLOROBENZILATE	39460	39461	79007		UG/L	UG/KG	MG/KG	HG/KG
875	HALATHION	39530	39531	39534		UG/L	UG/KG	HG/KG	HG/KG
876	DICOFOL (KELTHANE)	77902				UG/L	UG/KG	HG/KG	MG/KG
877	METHOMYL (LANNATE)	39051	82572	<b>B2570</b>		UG/L	UG/KG	HG/KG	HG/KG
878	ANTOR					UG/L	UG/KG	HG/KG	MG/KG
879	GUTHION	39580	39581	81802		UG/L	UG/KG	MG/KG	MG/KG
884	AMBUSH (PERMETHRIN)	79191				UG/L	UG/KG	MG/KG	MG/KG
885	BOLSTAR (SULPROFOS)	38715	38718	38719		UG/L	UG/KG	HG/KG	MG/KG
886	CYGON (DIMETHOATE)	39009	38460	38461		UG/L	UG/KG	HG/KG	MG/KG
887	DINOSEB (DNBP)	38780	38781	38782		UG/L	UG/KG	MG/KG	HG/KG
888	BPN	81290	<b>B2644</b>			UG/L	UG/KG	MG/KG	MG/KG
889	LORSBAN (CHLORPYRIFOS)	38932	38934	38744		UG/L	UG/KG	MG/KG	HG/KG
890	PYDRIN (FENVALERATE)	85757				UG/L	UG/KG	HG/KG	MQ/KG
891	SENCOR (METRIBUZIN)	81408	81409	82405		UG/L	UG/KG	MG/KG	HQ/KG
892	TREFLAN (TRIFLURALIN)	81284	81618	81652		UG/L	UG/KG	MG/KG	MG/KG
893	SEVIN	81816	81818	81899		UG/L	UG/KG	HG/KG	MG/KG
894	BALAN (BENEFIN)	39002				UG/L	UG/KG	HG/KG	HG/KG
895	TOTAL PCBS	39516	39526	39525		UG/L	UG/KG	HG/KG	MG/KG
896	DISYSTON	39011	,			UG/L	UG/KG	MG/KG	MG/KG
B97	PROMETON	39056	82402	82403	79698	UG/L	UG/KG	MG/KG	MG/KG
B98	PIPERONYL BUTOXIDE					UG/L	UG/KG	HG/KG	MG/KG
B99	DEET	38765	38768	38769		UG/L	UG/KG	HG/KG	MG/KG
900	PHORATE (THIMET)		<b>B1412</b>	39156		UG/L	UG/KG	MG/KG	MG/KG
901	DIOXATION	38783	38785	38786		UG/L	UG/KG	MG/KG	MG/KG
000	ORGANIC SCAN						•		
001	ETHYLENE GLYCOL	77023				UG/L	UG/KG	MG/KG	MG/KG
002	DIMETHYL MERCURY					UG/L	UG/KG	MG/KG	HG/KG
010	N-NITROSODIMETHYLAMINE	34438	34441	34442		UG/L	UG/KG	MG/KG	HG/KG
015	1,2-diphenylhydrazine/azobenzene	34346	34349	34350	79514	UG/L	UG/KG	MG/KG	MG/KG
020	BENZIDINE	39120	39121	34241	79553	UG/L	UG/KG	MG/KG	MG/KG
225	1,3-DICHLOROBENZENE	34566	34569	34570	79516	UG/L	UG/KG	MG/KG	HG/KG
330	1,4-DICHLOROBENZENE	34571	34574	34575	79517	UG/L	UG/KG	MG/KG	HG/KG
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NO.	TEST NAME	WATER	SED	<u>Fish</u>	WASTE	WATER	SED	FISH	WASTE
6035	1,2-DICHLOROBENSENE	34536	34539	34540	79511	UG/L	UG/KG	MG/KG	HG/KG
6040	BIS(2-CHLOROETHYL) ETHER	34273	34276	34277	79560	UG/L	UG/KG	MG/KG	HG/KG
6045	HEXACHLOROETHANE	34396	34399	34400	79636	UG/L	UG/KG	MG/KG	HG/KG
6050	BIS(2-CHLOROISOPROPYL) ETHER	34283	34286	34287	79561	UG/L	UG/KG	MG/KG	HG/KG
6055	N-NITROSODI-N-PROPYLAMINE	34428	34431	34432	79668	UG/L	UG/KG	MG/KG	HG/KG
6060	NITROBENZENE	34447	34450	34451	79672	UG/L	UG/KG	MG/KG	MG/KG
6065	HEXACHLOROBUTADIENE	39702	39705	34395	79634	UG/L	UG/KG	MG/KG	MG/KG
6066	2-METHYLNAPHTHALENE	77416	78868	79055		UG/L	UG/KG	MG/KG	MG/KG
6070	1,2,4-TRICHLOROBENZENE	34551	34554	34555	79507	UG/L	UG/KG	MG/KG	MG/KG
5075	NAPHTHALENE	34696	34445	34446	79670	UG/L	UG/KG	MG/KG	HG/KG
6076	4-CHLOROANILINE		78867	79053		UG/L	UG/KG	MG/KG	MG/KG
6080	BIS(2-CHLOROETHOXY) METHANE	3427B	34281	34282	79559	UG/L	UG/KG	MG/KG	HG/KG
5085	ISOPHORONE	34408	34411	34412	79646	UG/L	UG/KG	MG/KG	MG/KG
5090	HEXACHLOROCYCLOPENTADIENE (HCCP)	34386	34389	34390	79635	UG/L	UG/KG	MG/KG	HG/KG
5095	2-CHLORONAPHTHALENE	34581	34584	34585	79527	UG/L	UG/KG	MG/KG	HG/KG
5096	2-NITROANILINE		78299	79056		UG/L	UG/KG	HG/KG	HG/KG
100	ACENAPHTHYLENB	34200	34203	34204	79538	UG/L	UG/KG	MG/KG	HG/KG
105	ACENAPHTHENE	34205	34208	34209	79537	UG/L	UG/KG	HG/KG	MG/KG
110	DIMETHYL PHTHALATE	34341	34344	34345	79611	UG/L	UG/KG	HG/KG	HG/KG
111	DIBENSOFURAN	81302	75647	76619		UG/L	UG/KG	MG/KG	MG/KG
115	2,4-DINITROTOLUENE	34611	34614	34615	79524	UG/L	UG/KG	MG/KG	MG/KG
120	2,6-DINITROTOLUENB	34626	34629	34630	79525	UG/L	UG/KG	MG/KG	HG/KG
121	3-NITROANILINE	78300	78869	79057		UG/L	UG/KG	HG/KG	HG/KG
125	4-CHLOROPHENYL PHENYL ETHER	34641	34644	34645	79535	UG/L	UG/KG	HG/KG	HG/KG
126	4-NITROANILINE		78870	79058		UG/L	UG/KG	MG/KG	MG/KG
130	FLUORENE	34381	34384	34385	79626	UG/L	UG/KG	MG/KG	HG/KG
135	DIETHYL PHTHALATE	34336	34339	34340	79608	UG/L	UG/KG	MG/KG	MG/KG
140	N-NITROSODIPHENYLAMINE/DIPHENYLAMINE	34433	34436	34437	79669	UG/L	UG/KG	MG/KG	MG/KG
145	HEXACHLOROBENZENE (HCB)	39700	39701	34688	79633	UG/L	UG/KG	MG/KG	HG/KG
150	4-BROMOPHENYL PHENYL ETHER	34636	34639	34640	79534	UG/L	UG/KG	HG/KG	HG/KG
155	PHENANTHRENE	34461	34464	34465	79692	UG/L	UG/KG	MG/KG	MG/KG
160	ANTHRACENE	34220	34223	34224	79546	UG/L	UG/KG	HG/KG	MG/KG
165	DI-N-BUTYLPHTHALATE	39110	39112	34683	79599	UG/L	UG/KG	MG/KG	MG/KG

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EST			STORET	NOS.			REPORT	NG UNIT	'S
0.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
170	FLUORANTHENE	34376	34379	34380	79625	UG/L	UG/KG	MG/KG	MG/KG
175	PYRENE	34469	34472	34473	79702	UG/L	UG/KG	MG/KG	MG/KG
180	BENSYL BUTYL PHTHALATE	34292	34295	34296	79565	UG/L	UG/KG	MG/KG	MG/KG
185	BIS(2-RTHYLHEXYL) PHTHALATE	39100	39102	39099	79562	UG/L	UG/KG	MG/KG	HG/KG
190	BENZO(A) ANTHRACENE	34526	34529	34530		UG/L	UG/KG	MG/KG	MG/KG
195	CHRYBENE	34320	34323	34324	79592	UG/L	UG/KG	MG/KG	MG/KG
200	3,3'-DICHLOROBENZIDINE	34631	34634	34635	79530	UG/L	UG/KG	MG/KG	MG/RG
205	DI-N-OCTYLPHTHALATE	34596	34599	34600	79600	UG/L	UG/KG	MG/KG	MG/KG
210	BENZO(B AND/OR K) FLUORANTHENE	34711	34710			UG/L	UG/KG	MG/KG	MG/KG
215	BENZO(B AND/OR K) FLUORANTHENE	34711	34710			UG/L	UG/KG	HG/KG	HG/KG
220	BENZO-A-PYRENE	34247	34250	34251		UG/L	ng\ka	HG/KG	MG/KG
225	INDENO (1,2,3-CD) PYRENE	34403	34406	34407	79644	ng/r	UG/KG	MG/KG	MG/KG
230	DIBBNEO(A, H) ANTHRACENE	34556	34559	34560		UG/L	UG/KG	MG/KG	MG/KG
235	BENZO(GHI) PERYLENE	34521	34524	34525		UG/L	UG/KG	HG/KG	MG/KG
240	2-CHLOROPHENOL	34586	34589	34590	79528	UG/L	UG/KG	MG/KG	MG/KG
241	BENEYL ALCOHOL	77147	75212	76184		UG/L	UG/KG	MG/KG	MG/KG
242	2-METHYLPHENOL	77152				UG/L	UG/KG	MG/KG	MG/KG
243	(3-AND/OR 4-)METHYLPHENOL					UG/L	UG/KG	MG/KG	HG/KG
244	4-METHYLPHENOL	77146	78803	79146		UG/L	UG/KG	MG/KG	HG/KG
245	2-NITROPHENOL	34591	34594	34595	79529	UG/L	UG/KG	MG/KG	MG/KG
250	PHENOL	34694	34695	34468	79693	UG/L	UG/KG	HG/KG	MG/KG
255	2,4-DIMETHYLPHENOL	34606	34609	34610	79522	UG/L	UG/KG	MG/KG	MG/KG
256	BENZOIC ACID	77247	75315	76287		UG/L	UG/KG	MG/KG	MG/KG
260	2,4-DICHLOROPHENOL	34601	34604	34605	79521	UG/L	UG/KG	MG/KG	HG/KG
265	2,4,6-TRICHLOROPHENOL	34621	34624	34625	75920	UG/L	UG/KG	MG/KG	MG/KG
266	2,4,5-TRICHLOROPHENOL	77687	78401	79147		UG/L	UG/KG	HG/KG	MG/KG
270	4-CHLORO-3-METHYLPHENOL	34452	34455	34456	79682	UG/L	UG/KG	MG/KG	MG/KG
275	2,4-DINITROPHENOL	34616	34619	34620	79523	UG/L	UG/KG	MG/KG	MG/KG
280	2-METHYL-4,6-DINITROPHENOL	34657	34660	34661	79533	UG/L	UG/KG	MG/KG	MG/KG
285	PENTACHLOROPHENOL		78873	79157	79690	UG/L	UG/KG	MG/KG	MG/KG
290	4-NITROPHENOL	34646	34649	34650	79536	UG/L	UG/KG	MG/KG	MG/KG
291	2,3,4,6-TETRACHLOROPHENOL	77770	34721	78210		UG/L	UG/KG	MG/KG	MG/KG
500	DIOXIN/FURAN SCAN					NG/L	NG/KG	NG/KG	NG/KG
-55							-		-

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<u>rest</u>						REPORTING UNITS			
<u>, ON</u>	TEST NAME	WATER	<u>sed</u>	<u>Fish</u>	<u>wastr</u>	<u>water</u>	SED	FISH	<u>waste</u>
5705	1,3,5-TRICHLOROBENZENE	77614				UG/L	UG/KG	MG/KG	HG/KG
5710	1,2,3-TRICHLOROBENZENE	77613				ug/L	UG/KG	MG/KG	HG/KG
5715	FORMALDEHYDE	71880				UG/L	UG/KG	MG/KG	HG/KG
5716	ACETALDEHYDE	73528				UG/L	UG/KG	MG/KG	HG/KG
5760	4-CHLOROPHENOL	77296				UG/L	ug/kg	hg/kg	MG/KG
5765	BENZYLIC ACID					UG/L	UG/KG	MG/KG	MG/KG
57 <b>7</b> 0	BENZOPHENONE	77633				UG/L	UG/KG	MG/KG	HG/KG
5771	HONOBUTYL TIN					UG/L	UG/KG	MG/KG	HG/KG
5772	DIBUTYL TIN					ug/L	UG/KG	HG/KG	HG/KG
5773	TRIBUTYL TIN	30340				UG/L	UG/KG	HG/KG	HG/KG
5774	HONOPHENYL TIN					UG/L	UG/KG	MG/KG	HG/KG
5775	DIPHENYL TIN					UG/L	UG/KG	MG/KG	HG/KG
776	TRIPHENYL TIN					UG/L	UG/KG	hg/kg	HG/KG
780	2,3,7,8 TCDD(DIOXIN)	34675	34678	34679		NG/L	NG/KG	ng/kg	NG/KG
781	TETRACHLORODIBENZODIOXIN (TOTAL)					NG/L	NG/KG	NG/KG	NG/KG
782	PENTACHLORODIBENZODIOXIN (TOTAL)		30368	30344		NG/L	NG/KG	NG/KG	NG/KG
783	HEXACHLORODIBENZODIOXIN (TOTAL)		30376	30347		NG/L	NG/KG	NG/KG	NG/KG
784	HEPTACHLORODIBENZODIOXIN (TOTAL)		30379	30348		NG/L	NG/KG	NG/KG	NG/KG
785	OCTACHLORODIBENZODIOXIN					NG/L	NG/KG	NG/KG	NG/KG
786	2,3,7,8 TCDF (DIBENZOFURAN)	81302	75647	76619	79601	NG/L	NG/KG	NG/KG	NG/KG
787	TETRACHLORODIBENZOFURAN (TOTAL)					ng/l	NG/KG	NG/KG	ng/kg
788	PENTACHLORODIBENZOFURAN (TOTAL)					NG/L	NG/KG	NG/KG	NG/KG
789	HEXACHLORODIBENZOFURAN (TOTAL)					NG/L	NG/KG	NG/KG	NG/KG
790	HEPTACHLORODIBENSOFURAN (TOTAL)					NG/L	NG/KG	NG/KG	NG/KG
791	OCTACHLORODIBENZOFURAN					NG/L	NG/KG	NG/KG	NG/KG
792	TEQ(TOXICITY EQUIVALENT VALUE)					NG/L	NG/KG	NG/KG	NG/KG
795	BENZONITRILE	82297				UG/L	UG/KG	MG/KG	HG/KG
196	HMX (EXPLOSIVE)	82203				UG/L	UG/KG	MG/KG	MG/KG
197	RDX (EXPLOSIVE)	81364	81365			UG/L	UG/KG	MG/KG	MG/KG
198	THT (EXPLOSIVE)					UG/L	UG/KG	HG/KG	MG/KG
199	TETRYL (EXPLOSIVE)					UG/L	UG/KG	MG/KG	HG/KG
100	1,3-DINITROBENZENE					UG/L	DG/KG	MG/KG	HG/KG
101	1,3,5-TRINITROBENZENE					UG/L	UG/KG	HG/KG	MG/KG

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<u>.</u>	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE
00	VOA, PURGEABLE ORGANIC ANALYSES								
05	DICHLORODIFLUOROMETHANE	34668	34334	34335	79604	UG/L	UG/KG	MG/KG	MG/KG
10	TRICHLOROFLUOROMETHANE	34488	34491	34492	79719	UG/L	UG/KG	MG/KG	HG/KG
15	ACROLEIN	34210	34213	34214	79540	UG/L	UG/KG	HG/KG	MG/KG
20	ACRYLONITRILE	34215	34218	34219	79541	UG/L	UG/KG	MG/KG	MG/KG
5	CHLOROMETHANE	34418	34421	34422	79654	UG/L	UG/KG	MG/KG	MG/KG
0	BROMOMETHANE	34413	34416	34417	79653	UG/L	UG/KG	MG/KG	MG/KG
15	VINYL CHLORIDE	39175	34495	34693	79723	UG/L	UG/KG	MG/KG	MG/KG
0	CHLOROETHANE	34311	34314	34315	79588	UG/L	UG/KG	MG/KG	MG/KG
5	METHYLENE CHLORIDE	34423	34426	34427	79662	UG/L	UG/KG	MG/KG	MG/KG
0	1,1-DICHLOROETHENE(1,1-DICHLOROETHYLENE)	34501	34504	34505	79505	UG/L	UG/KG	MG/KG	HG/KG
1	ACETONE	81552	75059	76031	79539	UG/L	UG/KG	MG/KG	MG/KG
2	CARBON DISULFIDE	77041	78544			UG/L	UG/KG	MG/KG	MG/KG
•	VINYL ACETATE	77057				UG/L	UG/KG	HG/KG	HG/KG
5	1,1-DICHLOROETHANE	34496	34499	34500	79504	UG/L	UG/KG	MG/KG	MG/KG
5	CIS-1, 2-DICHLOROSTHENE	77093				UG/L	UG/KG	MG/KG	HG/KG
,	2,2-DICHLOROPROPANE	77170				UG/L	UG/KG	HG/KG	MG/KG
•	METHYL ETHYL KETONE	81595	75078	76050	79658	UG/L	UG/KG	MG/KG	HG/KG
)	BROMOCHLOROMETHANE	77297				UG/L	UG/KG	HG/KG	MG/KG
)	TRANS-1, 2-DICHLOROETHENE	34546	34549	34550	79515	UG/L	UG/KG	HG/KG	MG/KG
1	1,2-DICHLOROETHENE (TOTAL)					UG/L	UG/KG	HG/KG	MG/KG
5	CHLOROFORM	32106	34318	34319	79589	UG/L	UG/KG	HG/KG	MG/KG
Ó	1,2-DICHLOROSTHANE	32103	34534	34535	79512	UG/L	UG/KG	HG/KG	MG/KG
5	1,1,1-TRICHLOROETHANE	34506	34509	34510	79500	UG/L	UG/KG	MG/KG	MG/KG
6	1,1-DICHLOROPROPENE	77168				UG/L	UG/KG	HG/KG	MG/KG
0	CARBON TETRACHLORIDE	32102	34299	34300	79582	UG/L	UG/KG	MG/KG	MG/KG
5	BROHODICHLOROMETHANE	32101	34330	34331	79603	UG/L	UG/KG	MG/KG	MG/KG
5	METHYL ISOBUTYL KETONE		75169	76141		UG/L	UG/KG	MG/KG	HG/KG
0	1,2-DICHLOROPROPANE	34541	34544	34545	79513	UG/L	UG/KG	MG/KG	MG/KG
1	DIBROMOMETHANE	81522	78756	• • • • •		UG/L	UG/KG	MG/KG	MG/KG
5	TRANS-1,3-DICHLOROPROPENE	34699	34697	34698		UG/L	UG/KG	MG/KG	MG/KG
0	TRICHLOROETHENE (TRICHLOROETHYLENE)	39180	34487	34692	79718	UG/L	UG/KG	MG/KG	MG/KG

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TEST		· -	STORET	NOS.			REPORTING UNITS			
NO.	TEST NAME	WATER	SED	FISH	WASTE	WATER	SED	FISH	WASTE	
			-							
7105	BENZENE	78124	34237	34238	79551	UG/L	UG/KG	MG/KG	HG/KG	
7110	DIBROMOCHLOROMETHANE	34306	34309	34310		UG/L	UG/KG	HG/KG	MG/KG	
7115	1,1,2-TRICHLOROETHANE	34511	34514	34515	79503	UG/L	UG/KG	HG/KG	MG/KG	
7120	CIS-1,3-DICHLOROPROPENE	34704	34702	34703		UG/L	UG/KG	MG/KG	MG/KG	
7125	2-CHLOROETHYLVINYL ETHER	34576	34579	34580	79526	UG/L	UG/KG	MG/KG	MG/KG	
7130	BROHOFORM	32104	34290	34291	79563	UG/L	UG/KG	MG/KG	MG/KG	
7131	Brohobenzenz	81555				UG/L	UG/KG	HG/KG	HG/KG	
7135	1,1,2,2-TETRACHLOROETHANE	34516	34519	34520	79501	UG/L	UG/KG	MG/KG	MG/KG	
7140	TETRACHLOROETHENE (TETRACHLOROETHYLENE)	34475	34478	34479	79709	UG/L	UG/KG	HG/KG	MG/KG	
7141	1,3-DICHLOROPROPANE	77173				UG/L	UG/KG	HG/KG	MG/KG	
7142	METHYL BUTYL KETONE					UG/L	UG/KG	MG/KG	MG/KG	
7145	TOLUENE	78131	34483	344B4	79715	UG/L	UG/KG	MG/KG	MG/KG	
7150	CHLOROBENZENE	34301	34304	34305	79586	UG/L	UG/KG	MG/KG	HG/KG	
7151	1,1,1,2-TETRACHLOROETHANE	77562				UG/L	UG/KG	MG/KG	MG/KG	
7155	ETHYL BENZENE	34371	34374	34375	79624	UG/L	UG/KG	MG/KG	MG/KG	
7156	(M- AND/OR P-)XYLENE					UG/L	UG/KG	MG/KG	HG/KG	
7157	O-XYLENE	77135		79112		UG/L	UG/KG	MG/KG	MG/KG	
7158	STYRENE	77128	75192	76164	79707	UG/L	UG/KG	MG/KG	MG/KG	
7160	M-XYLENE					UG/L	UG/KG	MG/KG	MG/KG	
7165	OGP-XYLENE (MIXED)		,			UG/L	UG/KG	MG/KG	MG/KG	
7170	TOTAL XYLENES	81551	45510	45511	79724	UG/L	UG/KG	MG/KG	MG/KG	
7175	1,2,3-TRICHLOROPROPANE	77443				UG/L	UG/KG	HG/KG	MG/KG	
7180	O-CHLOROTOLUENE					UG/L	UG/KG	MG/KG	HG/KG	
7185	H-CHLOROTOLUENE					UG/L	UG/KG	MG/KG	MG/KG	
7190	P-CHLOROTOLUENE					UG/L	UG/KG	MG/KG	MG/KG	
7195	1,3-DICHLOROBENZENE	34566	34569	34570	79516	UG/L	UG/KG	MG/KG	MG/KG	
7200	1,4-DICHLOROBENZENE	34571	34574	34575	79517	UG/L	UG/KG	MG/KG	HG/KG	
7205	1,2-DICHLOROBENZENE	34536	34539	34540	79511	UG/L	UG/KG	HG/KG	HG/KG	
7720	1,2-DIBROMOETHANE (EDB)	77651				UG/L	UG/KG	MG/KG	HG/KG	
7725	EPICHLOROHYDRIN					UG/L	UG/KG	MG/KG	HG/KG	
7730	DIMETHYLAMINE	77003				UG/L	UG/KG	HG/KG	HG/KG	
7732	METHANE					UG/L	UG/KG	MG/KG	MG/KG	
7733	ISOPROPYL ETHER					UG/L	UG/KG	MG/KG	MG/KG	

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<u>o.</u>	TEST NAME	WATER	<u>SED</u>	FISH	WASTE	WATER	SED	FISH	WASTE
735	PROPYLENE GLYCOL DINITRATE					UG/L	UG/KG	MG/KG	MG/KG
005	CHLORINE RESIDUAL	50060				MG/L	MG/KG	HG/KG	MG/KG
010	DISSOLVED OXYGEN(WINKLER)	00300			•	MG/L	•	•	•
011	DISSOLVED OXYGEN (ELECTRODE)	00299				MG/L			
015	FLOW '	50050				MGD			
020	PH(FIELD)	00400				su	នប	su	BU
025	TEMPERATURE	00010				DEG C			DEG C
999	<b>♦ MOISTURE</b>		70320	70320	70320			•	•

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#### 8. SAMPLE COLLECTION AND HANDLING

#### 8.1. <u>Sample Collection - Water</u>

- 8.1.1. Water samples should be collected using standard field sampling techniques consistent with the parameter being determined. Sampling procedures are followed that minimize the possibility of sample adulteration by either the sample collector or sampling device. Field sample collection procedures are detailed in the, Environmental Compliance Branch, Standard Operating Procedures and Quality Assurance Manual.
- 8.1.2. Sample Containers and Sample Preservation. Containers and preservation techniques used must be consistent with the recommendations contained in Table 8-1.
  - 8.1.2.1. Selection of sample container types and preservation techniques are further guided by the method being applied.

    Additional guidance is available in references, e.g., Standard Methods for the Examination of Water and Wastewater, ASTM, Book of Standards, Volume 11.01 and 11.02 and EPA Methods for Chemical Analyses of Water and Waste.
  - 8.1.2.2. Sterilize bacteriological sample bottles at 121  $^{\circ}$ C for 15 minutes and select one bottle per batch to be checked for sterility by adding 25 ml of a sterile non-selective broth to the bottle. Incubate at 35 $^{\circ}$   $\pm$  0.5 $^{\circ}$ C for 24 hours, and check for growth.
  - 8.1.2.3. Samples must be accompanied by proper identification, e.g., tags, labels, and chain-of-custody forms. Sample source, date of collection, time of collection, and analysis required must be provided.
  - 8.1.2.4. Laboratory pure water blanks are prepared containing the preservative for each type of sample collected, such as metals, nutrients, phenols, etc. The same preservative is used for both blanks and samples. The blanks are then analyzed along with the samples for the constituents of interest.

#### 8.2. Sample Handling - Water

- 8.2.1. Handling of samples must be done in a manner that both insures the integrity of the sample and minimizes sample alteration. Sample custody is handled according to the procedures outlined in Section 3 of this document.
- 8.2.2. Environmental samples for bacterial analyses should be maintained in a wet-iced condition and should be analyzed within six hours after collection. Potable water samples must be analyzed within 30 hours after collection.

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8.2.3. When sample are not analyzed within the recommended holding time, a notation of this will be made in the final data report.

8.2.4. Intralaboratory sample control and handling is the responsibility of a project analyst. A project analyst is assigned to a project or group of samples at the time of request for analyses.

#### 8.3. Sample Collection and Handling - Other Substrates

- 8.3.1. Air, sediment, sludge, plant, and animal tissue samples, should be collected using techniques consistent with the parameter being determined and with the recommendations contained in Table 8-1. Sampling procedures are followed that minimize the possibility of sample adulteration either by the sample collector or sampling device.
- 8.3.2. Sediment and sludge samples for organic analyses must be collected in glass containers with Teflon or aluminum-foil-lined caps. Samples must be maintained at 4°C and analysed as soon as possible after collection. Sediment samples for extractable organic analyses must be in quart containers. Samples for VOA analyses must be in 40 ml VOA vials or 4 or wide mouth jars.
- 8.3.3. Sediment and sludge samples for nutrient and metal analyses should be collected in plastic containers and cooled.
- 8.3.4. Sediment and sludge samples for microbial analyses must be collected aseptically and placed in sterile containers. The sample should be maintained on ice or in a refrigerated condition. Analysis should be initiated within eight hours of collection.
- 8.3.5. Tissues from specific organs of fish or whole fish specimens should be frozen immediately after collection.
  - 8.3.5.1. If organic analyses are to be performed on fish tissue, the tissue should be wrapped in aluminum foil (shiny side out) prior to freezing. For metal analyses, plastic containers are acceptable.
  - 8.3.5.2. Fish requiring microbial analyses should be placed in plastic bags and maintained on ice or refrigerated until analysis. Analysis should be initiated as soon as possible after sample collection.

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# TABLES

# SAMPLE COLLECTION AND HANDLING

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rameter	Container	Preservative	Time	<u>Type</u>	Reference
ncentrated Waste Samples					
ganic Compounds- tractable and sticide/PCBs	8-oz. widemouth glass with Teflon liner	None	14 days	G or C	*
ganic Compounds- rgeable (VOA)	120-mL VOA container with Teflon lined Septusealed caps	None m	14 days	G or C	λ
tals and Other organic Compounds	8-oz. widemouth glass with Teflon liner	None	ASAP	G or C	λ
Toxicity	8-oz. widemouth glass with Teflon liner	None	ASAP — NS	G or C	В
ash Point and/or at Content	8-oz. widemouth glass with Teflon liner	None	ASAP - NS	G	В
sh Samples					
yanic Compounds	Wrap in aluminum foil (Shiny side out)	Freeze	ASAP	G or C	
als and Other organic Compounds	Place in plastic zip- lock bag	Freeze	ASAP	G or C	

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<u>cameter</u>	Container	Preservative	Holding Time	Permissible Sample Type	Reference
er - Low to Medium Concentr	ation Samples				
kalinity	500-ml or 1-liter poly-1 ethylene with polyethylene or polyethylene lined closure	Cool, 4°	14 days	G or C	C
dity	500-ml or 1-liter poly-1 ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	14 days	G or C	c
teriological	250-ml glass with glass closure or plastic capable of being autoclaved	Cool,4°C	6 hrs.	G	c
tic Bioassay	1-gal. amber glass (not solvent rinsed)	Cool, 4°C	48 hrs.	G or C	D
chemical Oxygen and (BOD)	1/2-gal. polyethylene ¹ with polyethylene closure	Cool, 4°C	48 hrs.	G or C	c
oride	500-ml or 1-liter poly-1 sthylene with polyethylene or polyethylene lined closure	None	28 days	G or C	c
orine Residual	In-situ, beaker or bucket	None	Analyze Immediately	g r	c
or ः	500-ml or 1-liter poly-1 ethylene with polyethy- lene or polyethylene lined closure	Cool, 4°C	48 hre.	G or C	C

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<u>rameter</u>	Container	Preservative	Holding Time	Sample Type	Reference
ter - Low to Mediu	m Concentration Samples (Co	ntinued)			
nductivity	500-ml or 1-liter poly-1 ethylene with polyethy- lene or polyethylene lined closure	Cool, 4°C	28 days (determine on site if possible)	G or C	c
comium, Hexavalent	1-liter polyethylene with polyethylene closure	Cool, 4°C	24 hrs.	G	c
ın <b>ide</b>	1-liter or 1/2-gallon polyethylene with poly- ethylene or polyethylene lined closure	Ascorbic Acid ^{2,3} Sodium Hydroxide, pH >12 Cool, 4°C	14 days	G	c
solved Oxygen obe)	In-situ, beaker or bucket	None	Determine On Site	G	c
solved Oxygen nkler)	300-ml glass, BOD bottle	Fix on site, store in dark	8 hrs. (determine on site if possible	G >)	C
Toxicity	1-gal. glass (amber) with Teflon liner	Cool, 4°C	ASAP - NS	G or C	B
pr <b>ide</b>	1-liter polyethylene or 1/2-gal. polyethylene with polyethylene or polyethy lene lined closure	None	28 days	G or C	<b>.</b>

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rameter	<u>Container</u>	Preservative	Holding Time	Permissible Sample Type	Reference
ter - Low to Hedi	um Concentration Samples (Co	ntinued)	:		
rdness	500-ml or 1-liter poly- ethylene with polyethy- lene or polyethylene lined closure	50% Nitric ² Acid, pH <2	6 months	G or C	c
S	500-ml or 1-liter poly-1 ethylene with polyethy- lene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	c
tale	1-liter polyethylene with polyethylene lined closure	50% Nitric ² Acid, pH <2	6 months	G or C	<b>c</b>
tals, Dissolved	1-liter polyethylene with polyethylene lined closure	Filter-on-site ² 50% Nitric Acid, pH <2	6 months	G	c
trients ⁴	1-liter polyethylene or 1/2-gal. polyethylene with polyethylene or polyethylene lined closure	50% Sulfuric ² Acid, pH <2 Cool, 4°C	28 days	G or C	c
L and grease	1-liter widemouth glass with Taflon lined cap	50% Sulfuric ² Acid, pH <2 Cool, 4°C	28 days	a	c

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# RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Preservative	Holding Time	Sample Type	Reference
Mater - Low to Medium C	oncentration Samples (Con	tinued)			
Organic Compounds - Extractable and Pesticide Scan					C
No Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon lined cap	Cool, 4°C	47 days ⁵	G or C	A or C
Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon lined cap	Add 3 ml 10% sodium thiosulfate per gallon Cool, 4°C	47 days ⁵	G or C	A or C
rganic Compounds - Purgeable (VOA)	:				
o Residual Chlorine Presènt	3 40-ml vials with Teflon lined septum sealed caps	4 drops 1+1 hydrochloric acid, Cool, 4 C	14 days	G	A or C
o Residual Chlorine Present	3 40-ml vials with Teflon lined septum sealed caps	Cool, 4°C	7 days	G .	A or C
Baidual Chlorine Present	3 40-ml vials with Teflon lined septum sealed caps	Footnote 6	14 days	G	A or C
rganic Compounds - pecified and asticides (Non- tiority Pollutants and as Herbicides)	1-gal. glass (amber) or 2 1/2-gal. glass (amber) with Teflon lined closure	Footnote 7	47 days ⁷	G or C	A or C
•		mahla 0 1 4	4.43	· · · · · · · · · · · · · · · · · · ·	

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# RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

ameter	<u>Container</u>	Preservative	Holding Time	Permissible Sample Type	Reference
er - Low to Medi	um Concentration Samples (Co	ntinued)			
anic Halides — al (TOX)	250-ml amber glass with Teflon lined septum closure	Cool, 4°C	ASAP — N8	<b>G</b>	A or E
	In-situ, beaker or bucket	None	Analyze Immediately	G	C
enols	1-liter amber glass with Teflon lined closure	50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	<b>G</b>	C
ephate-Ortho	500-ml or 1-liter poly- ethylene with polyethy- lene or polyethylene lined closure	Filter-on-site Cool, 4°C	48 hrs.	G	С
sphorus, Total solved	500-ml or 1-liter poly- ethylene with polyethy- lene or polyethylene lined closure	Filter-on-site 50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	G	C.
ids, Settleable	1/2-gal. polyethylene with polyethylene closure	Cool, 4°C	48 hrs.	G or C	C
ids (Total and pended, etc.)	500-ml or 1-liter poly-1 ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	7 days	G or C	c
fates	500-ml or 1-liter poly-1 ethylene with polyethy- lene or polyethylene lined closure	Cool, 4°C	28 days	G or C	c
		Table 8-1	(contd)	<u> </u>	

Table 8-1 (contd)

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<u>arameter</u>	<u>Container</u>	<u>Preservative</u>	Holding Time	Permissible Sample Type	Reference
ater - Low to Hedia	um Concentration Samples (Co	ontinued)			
ulfides	500-ml or 1-liter poly-2 ethylene with polyethy- lene or polyethylene lined closure	2 ml Einc Acetate ² Conc. Sodium Hydroxide to pH >9 Cool, 4°C	7 days	G	c
emperature	In-situ, beaker or bucket	None	Determine On Site	G	c
urbidity	500-ml or 1-liter poly-1 ethylene with polyethy- lene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	c
oil, Sediment or Sl	udge Samples - Low to Hedi	m Concentration			
. P. Toxicity	8-oz. widemouth glass with Teflon\ lined closure	Cool, 4°C	ASAP - NS	G or C	В
etals	8-oz. widemouth glass with Teflon lined closure	Cool, 4°C	6 months	G or C	λ
itrients icluding Nitrogen, iosphorus, iemical Oxygen imand	500-ml polyethylene with polyethylene closure or 8 oz. widemouth glass with Teflon lined closure	Cool, 4°C	ASAP	G or C	<b>A</b>
'ganics - 'tractable	8-os. widemouth glass with Teflon liner	Cool, 4°C	ASAP	G or C	A

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<u>rameter</u>	Container	Preservative	Holding Time	Sample Type	Reference
<u>il, Sediment or S</u>	ludge Samples - Low to Medium C	oncentrations (Cor	ntinued)	•	
ganics - rgeable (VOA)	4-oz. (120 ml) widemouth glass with Teflon liner	Cool, 4°C	ASAP	G or C	A
her Inorganic mpounds - cluding Cyanide	500-ml polyethylene with polyethylene closure or 8-oz. wide mouth glass with Teflon lined closure	Cool, 4°C	ASAP	G or C	A
nicipal Sludge -	Low to Medium Concentrations				
ganics - tractable & sticide/PCBs	<pre>0 - 30% Solids 1- gal. amber glass or 4 qt wide mouth bottle (depending on consistency)</pre>	Cool, 4°C	47 daya ⁵	G or C	P
	with Teflon lined cap > 30% Solids 8-os widemouth glass jar with Teflon lined cap	Cool, 4°C	47 days ⁵	G or C	F
janics - :geables (VOA)	O - 30% Solids 3 40-mL VOA vials with Teflon lined septum sealed caps or 1 120-mL	4 drops 1+1 HCl acid, Cool, 4°C	14 days	G or C	<b>P</b>
	VOA if consistency requires is > 30% solids 120-mL VOA vial with Teflon lined septum sealed cap	Cool, 4°C	14 days	G or C	r

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### RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

#### Abbreviationer

G = Grab

C = Composite

ASAP = As Soon As Possible

NS = Not Specified

#### ootnotes:

- .. Use indicated container for single parameter requests, 1/2-gallon polyethylene container for multiple parameter requests except those including BOD, or 1-gallon polyethylene container for multiple parameter request which include BOD.
- . Hust be preserved in the field at time of collection.
- . Use ascorbic acid only if the sample contains residual chlorine. Test a drop of sample with potassium iodide-starch test paper; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.
- . May include nitrogen series (ammonia, total Kjeldahl nitrogen, nitrate-nitrite), total phosphorus, chemical oxygen demand and total organic carbon.
- . Samples must be extracted within seven days and extract must be analyzed within 40 days.
- . Collect the sample in a 4 oz. soil VOA container which has been pre-preserved with four drops of 25 percent ascorbic acid solution. Gently mix the sample and transfer to a 40 ml VOA vial that has been prepreserved with four drops 1+1 HCl, cool to 4°C.
- See Organic Compounds Extractable (page 4 of 8). The Analytical Support Branch should be consulted for any special organic compound analyses in order to check on special preservation requirements and or extra sample volume.

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### RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

#### ferences:

US-EPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Office of Solid Waste and Emergency Response, Washington, DC, Nov. 1986.

US-EPA, Test Methods for Evaluating Solid Waste, SW-846, Office of Solid Wastes, Washington, DC, 1982.

40 CFR Part 136, Federal Register, Vol. 49, No. 209, October 26, 1984.

US-EPA, Region IV, Environmental Services Division, "Ecological Support Branch, Standard Operating Procedures Hanual," latest version.

EPA Interim Method 450.1, "Total Organic Halide," US-EPA, ORD, EMSL, Physical and Chemical Methods Branch, Cincinnati, Ohio, November 1980.

US-EPA, <u>Analytical Methods for the National Sewage Sludge Survey</u>, Office of Water Regulations and Standards, Washington, DC, Aug. 1988.

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#### 9. SAMPLE RECORDS AND DATA HANDLING

9.1. Sample accountability through the analytical process can be divided into three major elements: (1) initial sample logging; (2) data acquisition, and (3) documentation/storage. The laboratory location, i.e., field or central, and the analyses requested will dictate the nature and location of the sample and data records. In addition to the procedure discussed in Section 3 of this manual, the following sections outline current sample and data documentation procedures.

#### 9.2. CHEMICAL SAMPLE LOGGING

#### 9.2.1. Field Logging

9.2.1.1. Samples received at a field laboratory with accompanying identification are logged into the field sample logbook. Samples are assigned consecutive log numbers. These numbers are assigned by the sample custodian.

#### 9.3. Central Laboratory Sample Logging

- 9.3.1. Samples received at the central laboratory with accompanying identification are logged into the SAAMS. Samples are assigned consecutive log numbers and logged as described above.
- 9.3.2. Also contained in the SAAMS is a description of the disposition of every log number used, whether in the field or central laboratory.

#### 9.4. Chemical Data Handling

#### 9.4.1. General

- 9.4.1.1. All raw analytical and instrument control data generated in the laboratory are entered into bound data books or kept as strip charts, or in instrument computer hardcopy, tape, or disk.
- 9.4.1.2. Information contained in these data logbooks includes the following: parameter, project, date of analysis, analyst, sample log number, all calibration data, all readout data, calculation, final concentration, and quality control data.
- 9.4.1.3. Final results of all analyses are provided in a standard computerized report format and forwarded to the requester with cover memorandum. The analytical data is ready for automatic transfer into the Storet system if Storet station location data is available. Standard remarks can be used with reported data to alert the user to some specific condition that affects the data. A list of these remarks appears in Section 10.
- 9.4.2. More specific information on data handling is contained in Section 9.6.2.1 and Sections 10 and 11.

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#### 9.5. Microbiology Sample Logging

9.5.1. Samples received with accompanying identification are assigned consecutive log numbers and are recorded in the Microbiology Sample and Analysis Log. Sample information recorded in the log includes: Sample log number, station identification, date and time collected, received and analyzed, signature of person delivering sample, the signature of analyst receiving the sample. Sample number, station identification, and data collected are subsequently transferred to a microbiology bench card (Form 9-1) for laboratory use.

#### 9.6. Microbiology Data Handling

- 9.6.1. All analytical results are recorded on a laboratory bench card (Form 9-1), and completed data are then transferred to the sample and analysis log.
- 9.6.2. Qualitative determinations, e.g., biochemical identifications, are recorded in the biochemical log. Information maintained in the logbook includes: sample log number, sample or isolate source, enrichment media used, selective media used, differential media used, biochemical test run, serological reactions, and organism identification.

#### 9.7. Computerized Analytical Data System

#### 9.7.1. Introduction

- 9.7.1.1. The "Sample and Analyses Management System" (SAAMS) is a computerized data storage and laboratory information management system. SAAMS is mainly utilized by the Analytical Support Branch (ASB), ESD, and maintained for the Branch by the Region IV Information Management Branch. The system was initiated in 1978 with a sample logging process and has since been expanded to complete laboratory data management system.
- 9.7.1.2. SAAMS is structured using the INFORM data base management system. INFORM is a very flexible, interactive system that integrates a variety of data processing functions within the structure of one high level language.
- 9.7.1.3. SAAMS is located on the DEC Microvax computer at the Environmental Research Laboratory in Athens, GA. All communication with the Microvax is through terminals and/or PC's located in offices of the Environmental Services Division. These terminals are either direct wired or operated through telephone couplers.
- 9.7.1.4. Normally, every night a batch input stream, named AUTOREPT, is submitted which executes 40 INFORM programs that produce all reports, data sheets and backlogs. The details of AUTOREPT will be discussed in each of the following sections.

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9.7.1.5. Figure 9-1 presents a general flow of data in SAAMS.

#### 9.7.2. System Description

#### 9.7.2.1. Project Logging

- 9.7.2.1.1. All Analytical projects, when initiated by the requester, are logged into a data base named PROJLOG. Project numbers are assigned by ASB starting with FY-001 at the beginning of each fiscal year (e.g., FY=89, 90, etc). This file contains all identification information for the project such as: project number, name of project, location, date project to be conducted, requester and program element, account number, time accounting information, etc.
- 9.7.2.1.2. If the samples from the project are to be analyzed by the Contract Laboratory Program (CLP), the contract laboratory name and case number are identified.
- 9.7.2.1.3. Each time AUTOREPT is run, computed information is copied into fields in PROJLOG, such as analytical completion dates. Also, when AUTOREPT runs, information in the receipt date field and the Account No. field are compared to the same fields in the Environmental Compliance Branch's (ECB) Project Log. If they differ, the information in the engineering project log is copied into PROJLOG.

#### 9.7.3. Sample Logging

- 9.7.3.1. All samples to be analyzed or tracked by the ASB are logged into the sample logging data base, NEWLOG, when received. The samples are numbered in chronological order.
- 9.7.3.2. Data entered into NEWLOG identifies and describes each sample, the tests required, and the test numbers. Test numbers are maintained in a file named 83.Zebra. A printout of the sample data log sorted in sample number order by project is also produced. The blank data sheets and log printout are given to the chemists involved in the project. A copy of the sample data log printout is filed in the project file and another copy is sent to the requester along with his copy of the custody record.

#### 9.7.4. Analytical Data Processing

- 9.7.4.1. All analysis results are entered into the analytical results data bases.
- 9.7.4.2. When data are entered manually and accepted, they are flagged in a manner that AUTOREPT will produce a proof copy of the data which the analyst checks against the data sheet that he filled out. Any errors detected are corrected and the data is verified. AUTOREPT will next produce a final production copy of the data sheet that will be sent to the requester. A job named MOVEMERGE can be submitted at any time. This set of programs will

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move all completed verified data from the different data bases into identically structured data bases in another account. The ECB can access this account and summarize and tabulate data from it. However, data cannot be added, deleted or modified by the ECB.

9.7.4.3. The majority of all data is transmitted electronically to the Microvax, with the above proofing steps conducted at the analyst's work station.

#### 9.7.5. Other ADP Operations

9.7.5.1. Quality Control Data. Data bases (QC.FILE and SURROGATE.FILE) are available for entering, storing and summarizing precision and accuracy data generated during sample analysis. This includes percent RSD, matrix spike recovery data, surrogate spike recovery data, results of reference sample analyses, etc. Entry and varification programs are available for this QC operation.

Summary programs are available that can be applied at command to the entire QC data base or to any portion of the data base required. Data bases for summary can be made from the main data base using any of the input fields.

- 9.7.5.2. Sample Custody Information. Sample custody information, such as custody room check-out and check-in information, sample disposal information, etc., is kept in a custody file (CUSTODY. ROOM.LOG). Information in this file and the sample log file can be combined to give a complete documentation of chain-of-custody for all samples.
- 9.7.5.3. Time Accounting Information. A data base named LABTIME, divided into fiscal years, is maintained for storage and manipulation of personnel time. All personnel time is entered by employee, pay period, activity, account number and program element. Different INFORM processes can be applied to this file for time accounting purposes.
- 9.7.5.4. Accounting Reports. At any time a batch job named ACTGREPT can be submitted which produces the following accounting reports:
  - o Sample Counter Listing of number of samples received by type, program element, and whether analyses were performed by EPA or a contract lab.
  - o Analysis Counter Listing of analyses by parameter, sample type, and whether analyses were conducted by EPA or contract laboratory.
  - o Determination Reports Listing of number of determinations of each parameter by sample type and

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whether analysis were conducted by EPA or a contract laboratory.

o Total Accounting Report - Listing of analyses within each program element by parameter, sample type and whether analyses were conducted by EPA or contract laboratory.

- 9.7.5.5. Analytical Backlogs. Each time that AUTOREPT is submitted, several types of backlogs are produced that give information on incomplete projects and projects not yet received. These are used for tracking progress of samples being analyzed and for planning analysis of samples not yet received.
  - o Chemistry Backlog inhouse samples: List projects in chronological order with name, project number, program element, requester, receipt data (actual or projected), projected completion date, number of samples scheduled to be received or number of samples received broken down into analytical categories (inorganics, VOA, extractable organics, pesticides, metals, and EP).
  - o Chemistry Backlog contractor samples: Same information as above except for contract samples.
  - o Detail Backlog. Listing of all required analyses, by parameter test code, sample type, project number and sample number. The listing separates inhouse analyses from contractor analyses.

The backlogs are updated each time AUTOREPT is run and are currently updated based on sample receipt, input and verification of analytical data.

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# **FORMS**

### SAMPLE RECORDS AND DATA HANDLING

SECTION 9

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#### MICROBIOLOGY BENCH CARD

#### BACTERIAL INDICATOR ORGANISMS OF POLLUTION

STATION		BENCH NO.					•
LOCATI	ои	<del></del>					
		DATE		HOURS			
COLLEC	TED			ANA	LYST		
EXAMIN	ED			• : • —			
		MPN					
DiL.	PRESUMPTIVE LTB 35 C		CONFIRMED B.G.L.B 35 C		EC 44.5 C	VEVmns via MTT men	
						MEMBRANE FILTER	
	24 hrs.	48 hrs.	24 hrs.	48 hrs.	24 hrs.	VOLUME FILTERED	COLONY
10							TOTAL COLIFORF
10							FECAL COLIFORMS
10							
						ORGANIS	MS PER 100 ML
10						TOTALS_	
						FECALS	
10						SALMONELL	λ
						<u></u>	
CONFIRMED MPN PER 100 ML. FECAL COLIFORM MPN PER 100 ML. REMARKS:							
				SEROTYPE (	s)		

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## FIGURES

### SAMPLE RECORDS AND DATA HANDLING

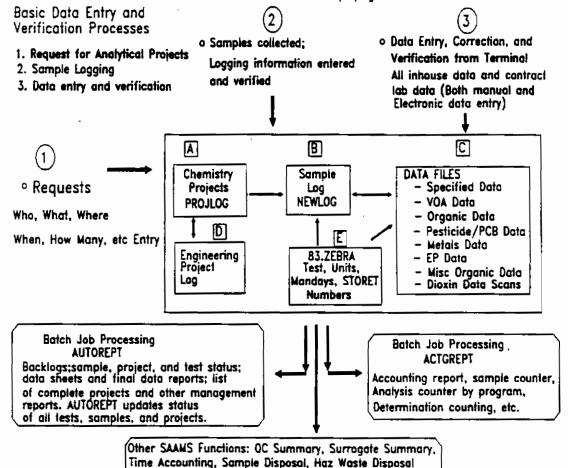
SECTION 9

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# US EPA REGION IV — ANALYTICAL SUPPORT BRANCH July 1990 "Sample and Analysis Management System" (SAAMS)

SAAMS is contained on a DEC Microvax employing INFORM software



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#### 10. ORGANIC ANALYSIS, PERFORMANCE QUALITY CONTROL AND ANALYTICAL OPERATION

10.1. Every element of environmental data acquisition, from sample collection to final data reporting, has associated with it degrees of error. The primary purpose of a total quality assurance program is the optimization of conditions whereby the introduction of error can be either precluded or substantially reduced. The operating procedures and quality control checks practiced in this laboratory and outlined in this manual are implemented to minimize the total error associated with data generation. No number can be affixed to total error; however, analytical performance is measurable and thus definable.

#### 10.2. General

- 10.2.1. During the course of generating data on samples for organic parameters, it is the policy of this Branch to apply the best laboratory practices, use approved methodology when mandated by regulation, use standardized methodology, if possible, when approved methodology is not applicable, fully document all operations associated with the generation of data and to meet certain quality requirements that will be designated in the following paragraphs.
- 10.2.2. Safety precautions associated with the safe handling of toxic chemicals, reagents, solutions and samples will be observed and regarded as a first order responsibility of the analyst. The analyst will take the necessary precautions to prevent exposure or harm both to himself and his fellow workers.

#### 10.3. Organic Methodology

10.3.1. Section 7 contains a listing of individual analytical methods used. Table 10-1 contains a listing of current analytical descriptors associated with these methods. These descriptors are used in sample vial labeling and file naming conventions in GC and GC/MS computer systems as appropriate.

#### 10.4. Sample Preparation of Semivolatile fraction and Pesticide fraction

#### 10.4.1. General Quality Control Requirements

- 10.4.1.1. All glassware and glass wool are rinsed sequentially with methanol, acetone, and the sample solvent, just prior to use.
- 10.4.1.2. A reagent blank is set up with each set of 20 or less samples when an extraction is performed or when simply putting a chemical waste sample in solution. Include all glassware and extraction equipment.
  - 10.4.1.2.1. Water Use Milli-Q water and all solvents and reagents in the reagent blank.

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- 10.4.1.2.2. Soil/Sediment/Tissue Use the appropriate amount of anhydrous sodium sulfate and all solvents and reagents.
- 10.4.1.2.3. Waste Use anhydrous sodium sulfate and all solvents and reagents.
- 10.4.1.3. Duplicate matrix spikes (spike two portions of a sample expected to contain no organics or low levels) and/or duplicate method spikes (media known to be organic free, i.e., Milli-Q water or for solids, clean sand and anhydrous sodium sulfate). Duplicate spikes are included with each set of 20 or less samples.
- 10.4.1.4. The spiking of samples is performed by an experienced analyst.
- 10.4.1.5. A gel permeation chromatograph(GPC) calibration standard consisting of corn oil, bis(2-ethylhexyl)phthalate, 4-nitrophenol, perylene, and sulfur must be passed through the GPC system prior to beginning cleanup of samples, once/month. This must be done more frequently after repacking the column. Adjust the collection volume to recover  $\geq$  85% of the bis(2-ethylhexyl)phthalate.

#### 10.4.2. General Extraction Protocols

- 10.4.2.1. Determination of percent moisture
  - 10.4.2.1.1. Sediment/Soil percent moisture must be determined on all samples unless otherwise specified.
  - 10.4.2.1.2. Waste sample Determine percent moisture if the sample is primarily heavily contaminated soil or a dry solid. This must be done in an oven located in a hood. Waste that is primarily a non aqueous liquid does not require a percent moisture determination.
- 10.4.2.2. Chlorinated water samples must be dechlorinated with sodium thiosulfate prior to extraction.
- 10.4.2.3. All water samples extracted for pesticide analyses from compliance sampling inspections(CSI) and toxic compliance sampling inspections(XCSI), and all water extracts with color must be passed through the alumina microcolumn.
- 10.4.2.4. If the final extract volume is greater than 1 mL, transfer at least 1 mL to a GC vial. The remainder is discarded. Never leave any extracts in volumetric flasks.
- 10.4.2.5. Labeling Laboratory Sample Containers
  - 10.4.2.5.1. One or more of the analytical descriptors (See Table 10-1) should be used as a suffix after each sample number recorded on the sample vial and in the extraction log (i.e. 10234SLS for a semivolatile low soil extract of sample 10234).

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- 10.4.2.5.2. Record the inclusive sample numbers on the blank, spike, and spike standard vial labels of the samples that were extracted with the blank and spike.
- 10.4.2.5.3. B Blank, include inclusive sample numbers after B (e.g. B5440-5461SLW)
- 10.4.2.5.4. S -Spike, include sample designation and sample number spiked after S (e.g. S12440P for pesticide spike of sample 12440)
- 10.4.2.5.5. X and Y To designate duplicate extractions.
- 10.4.2.5.6. R, R2, R3, etc. To designate when re-extractions are required, designate them with an "RX" depending on the number of re-extractions required.
- 10.4.2.5.7. A mark is placed on each sample vial to indicate the bottom of the meniscus when vialed.
- 10.4.2.5.8. The final extract volume is recorded on all vials and in the extraction log.

#### 10.4.3. Extraction Logbook

- 10.4.3.1. All pertinent information requested on the sheet will be properly recorded prior to submittal to the GC and or GC/MS chemists. See Forms 10-1, 10-2, 10-3.
- 10.4.3.2. List the blank and spike in the sample number column. Record the sample numbers, the blank and spike represent (example: blank 1411-25; spike 1411-25).
- 10.4.3.3. Record the extract volume on the sheet.
- 10.4.3.4. Record the designation for extract type after the sample number.
- 10.4.3.5. Record unusual occurrences during sample preparation, e.g., unusual appearance of sample, problems during extraction, losses of extract, precipitation and/or increase in viscosity during final evaporation, etc.
- 10.4.3.6. All calculations must be checked by a second person and the extraction sheet initialed.
- 10.4.3.7. Do not use "Liquid Paper" to correct any error. Put one line through the error with initials and date.

#### 10.4.4. Sample Vial Handling

10.4.4.1. Put all vials on one board that pertain to a project, and label the board with the project name. The chemist in charge

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of the extraction laboratory should check the labeling of all vials. Do not put two separate extraction batches on one board.

10.4.4.2. Sample vials and copies of the extraction sheet should be given to the chemist in charge of the pesticide or semivolatile analysis.

10.4.4.3. Include a surrogate standard solution with each set of samples. This solution should be at the same concentration as in the sample extracts.

#### 10.5. Surrogate Standards

10.5.1. A surrogate standard, a chemically inert compound not expected to occur in an environmental sample, is added to each sample just prior to extraction or purging. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the statistical acceptance limits.

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10.5.2. Following are the surrogate standards and the corresponding spike solution concentrations currently used by ASB:

Semivolatile-Base/Neutral	Sol'n Conc.	Spike Amt per Final Extract Volume
Nitrobenzene - d ₅ Terphenyl - d ₁₄	1000ng/uL 1000ng/uL	50uL/lmL
<u>Semivolatile-Acid</u>	Sol'n Conc.	Spike Amt per Final Extract Volume
2,4,6-tribromophenol phenol - d ₆	1000ng/uL 1000ng/uL	50uL/lmL
Volatiles-Water/Soil/Sed	Sol'n Conc.	Spike Amt per Final Purge Volume
toluene - d ₈ p-bromofluorobenzene dibromofluoromethane	25ng/uL 25ng/uL 25ng/uL	SuL/SmL
Volatiles-Air Canister	Sol'n Conc.	Spike Amt per Final Canister Volume
toluene - d ₈ p-bromofluorobenzene dibromofluoromethane	125ng/uL 125ng/uL 125ng/uL	10uL/12L
Organo-chlorine Pesticides and PCBS	Sol'n Conc.	Spike Amt per Final Extract Volume
dibutylchlorendate (DBC) 2,4,5,6 tetrachloro-	40ng/uL	25uL/1mL
meta-xylene (TCMX)	20ng/uL	
Phenoxy Herbicides	Sol'n Conc.	Spike Amt per Final Extract Volume
DCAA (2,4 -Dichlorophenyl- Acetic Acid)	20ng/uL	100uL/10mL
Organonitrogen/phosphate Pesticide	Sol'n Conc.	Spike Amt per Final Extract/Purge Volume
2-Nitro-m-xylene (NMX)	250ng/uL	50uL/1mL

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- 10.5.3. Calculation of Acceptance Limits (All calculations are performed by the laboratory SAAMS ADP System.)
  - 10.5.3.1. Calculate average recovery (R) and standard deviation (S), in percent recovery, for each surrogate standard from the entire data base.
  - 10.5.3.2. Values greater than 3 standard deviations are eliminated from the data base as outliers. The limits are then re-calculated as above.
  - 10.5.3.3. Calculate method performance criteria and define the performance of the laboratory for each surrogate standard being used.
  - 10.5.3.4. Calculate upper and lower control limits for method performance and surrogate standard recovery:

#### Semivolatiles and Pesticides:

Upper Control Limit (UCL) = R + 2 S

Lower Control Limit (LCL) = R - 2 S

#### Volatiles:

Upper Control Limit (UCL) = R + 3 S

Lower Control Limit (LCL) = R - 3 S

10.5.3.5. Surrogate limits are calculated annually.

#### 10.5.4. Analysis of Surrogates

- 10.5.4.1. Purgeable Organics All Samples and blanks are to be analyzed by GC/MS. The GC/MS analyst is responsible for calculating recovery and recording the data in the GC/MS logbook and transferring to the IBM PC data base using Datamaster.
- 10.5.4.2. Extractable Organics and Pesticides Most samples and blanks will be analyzed by GC/MS for extractable organics and by GC/EC or GC/Hall for pesticides. The analyst is responsible for recording the surrogate data on the computer logsheet. The pesticide data must be archived in SAAMS and the PC storage system. The semivolatile and volatile organics data must be transferred to the IBM PC data base using Datamaster.

Percent Surrogate Recovery = Q X 100

Q,

Where  $Q_d = Quantity$  determined by analysis

Q = Quantity added to the sample

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#### 10.5.5. Evaluation of Surrogate QC Data

10.5.5.1. Purywable Organics - If surrogate standard recovery of any one surrogate is out of limits in a blank or a sample, proceed with corrective action.

10.5.5.2. Extractable Organics - If two surrogates from the same sample pE fraction are out of limits or any surrogate is <10% recovery, proceed with corrective action. See below for action on blanks and matrix or method spikes.

10.5.5.3. Pesticide/PCB - Since GC/EC data is much more subject to interference than GC/MS, a secondary surrogate is to be utilized when sample interference is apparent. Dibutylchlorendate (DBC) is also subject to acid and base degradation. Therefore, two surrogate standards are added to each sample; DBC and 2,4,5,6-tetrachloro-meta-xylene (TCMX). DBC is the primary surrogate and should be utilized whenever possible. However, if DBC recovery is low or compounds interfere with DBC, then the TCMX should be evaluated for acceptance. Proceed with corrective action when both surrogates are out of limits for a sample. See below for action on blanks and matrix or method spikes.

10.5.5.4. At present there are no QC limits for the herbicide and organo-nitrogen/phosphorus surrogates.

#### 10.5.5.5. Corrective Action

10.5.5.5.1. Check for instrumental problem, make any corrections and redilute the extract if necessary, and then rerun the sample. This also applies to blanks and matrix or method spikes.

10.5.5.5.2. If no instrumental problems exist, the sample should be re-extracted and re-analyzed. However, if the sample data from the first analysis has to be reported, report out the data from the first analysis and flag the data with a "J". If surrogates from extractable and pesticide blanks exceed the above criteria, but one or more samples in the set have acceptable surrogate limits, evaluate the blanks carefully. Do the blanks still provide sufficient information to determine the presence of contaminants in the samples? Matrix or method spikes - since these are already prepared in duplicate, no re-extraction is required. If both duplicates are out, indicating a matrix effect, record matrix surrogate recovery data for both.

10.5.5.3. If the surrogates are still outside the acceptance limits after repurging or re-extraction, the data should be reported and flagged with a "J".

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10.5.6.1. All surrogate data must be transferred to the IBM PC using Datamaster or recorded on the Surrogate Recovery form and submitted to the Branch ADP Coordinator or downloaded directly to SAAMS. The only exception to this is surrogate data that is known to be in error; i.e. acid not added to water prior to water extraction; valve on GPC instrument leaking and causing cross contamination of samples; purge and trap system contaminated, etc. Also, do not record dibutylchlorendate data when pH is outside neutral range during extraction or cleanup.

#### 10.6. Internal Standards

10.6.1. An internal standard, a chemically inert compound not expected to occur in an environmental sample, is added to each sample just prior to instrumental analysis.

10.6.2. Following are the internal standards and the corresponding spike solution concentrations currently used by ASB:

<u>Semivolatile</u>	Sol'n Conc.	Amt per Final Extract Volume
1,4-Dichlorobenzene-d ₄ Naphthalene-d ₈ Acenaphthene-d ₁₀ Phenanthrene-d ₁₀ Chrysene-d ₁₂ Perylene-d ₁₂	1000ng/uL 1000ng/uL 1000ng/uL 1000ng/uL 1000ng/uL 1000ng/uL	10uL/lmL
Volatiles-Water/Soil/Sed	Sol'n Conc.	Spike Amt Per Final Volume
Difluorobenzene Chlorobenzene-d ₅ 1,4-Dichlorobenzene-d ₄	25ng/uL 25ng/uL 25ng/uL	SuL/SmL
Volatiles-Air Canister	Sol'n Conc.	Spike Amt Per Final Volume
Difluorobenzene Chlorobenzene-d ₅ 1,4-Dichlorobenzene-d ₄	125ng/uL 125ng/uL 125ng/uL	10uL/12L

#### 10.7. GC Analysis

#### 10.7.1. GC Logbook

10.7.1.1. Be sure all pertinent information requested on the sheet is properly recorded. See Form 10-11.

10.7.1.2. All analysts that participated in making dilutions and/or loading the auto-sampler must record their name. This includes analysts that add extracts at the end of the run.

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- 10.7.1.3. Record inclusive sample numbers for each blank and spike.
- 10.7.1.4. Record concentration of standard or the name of the standard (e.g., Red Mix VI).
- 10.7.1.5. Record all information that is on the sample vial.
- 10.7.1.6. Record all dilutions with dilution factor, times sign, and original volume (example: 10 X 1 mL or 10,000 X of 25 mL).
- 10.7.1.7. A copy of the logbook page should be made for each project file.
- 10.7.2. Procedure for analyzing samples and transmitting and storing data by using a PC and Nelson 2600 chromatography software.
  - 10.7.2.1. Set the PC for the correct time and date.
  - 10.7.2.2. Build a new method or use an existing one that will be suitable for the analysis to be done (e.g. Herb., PCB only, Pest. and PCB).
    - 10.7.2.2.1. Create a header page. Be sure to enter the project(s) name(s) and all analysts names or initials that made dilutions and/or loaded the auto-sampler.
    - 10.7.2.2.2. Edit operating parameters. Enter the variables needed to identify and quantify the compounds of interest (e.g. run time, noise threshold, offset in MV., external Std. report (y,n).
    - 10.7.2.2.3. Edit timed events. Timed events act as software switcher. Most cause an on/off change at a scheduled time during an analysis (eq. force baseline at peak start, Peak detection (on/off), force peak skim).
    - 10.7.2.2.4. Edit component names. Enter all compound names that are needed for the analysis and be sure to enter the test number for each compound. Enter parameters needed for each compound (e.g. retention time, window size, etc.)
    - 10.7.2.2.5. Give the method a suitable file name and store it in drive M:
    - 10.7.2.2.6. Download the method to the interface.
  - 10.7.2.3. Create an instrument header for each analysis. This should include: (1) instrument code(2) column type (3) mobile phase (4) operating conditions (5) carrier gas (6) column length (7) detector information.
  - 10.7.2.4. Create a sequence file for each GC run. Enter the number of samples and standards to be analyzed in the order of

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their injection. For each injection, a sequence should contain the following information:

- 10.7.2.4.1. Name of sample or standard. Record the media code (e.g. w/for water). Record inclusive sample numbers for each blank and spike and code for each standard (eg. RCDI). A sample name for a water extract would appear like: w/45225 or for water blank: w/45225-30/blk.
- 10.7.2.4.2. Name of the method to be used for the analysis.
- 10.7.2.4.3. Base name for the raw data files. This should include the drive designated for the analyst and a appropriate file name (e.g. V:MC).
- 10.7.2.4.4. Volume of sample injected.
- 10.7.2.4.5. Dilution factor (L < or = six digits).
- 10.7.2.4.6. Sample weight or volume.
- 10.7.2.4.7. Create an appropriate file name for the sequence and store in drive M: (eq. M:MC).
- 10.7.2.5. Set the acquire program to download the data from the interface to the PC during the GC run or wait to the end of the GC run.
- 10.7.2.6. Set the GC conditions for the run and then start the auto-sampler and the GC run.
- 10.7.2.7. After the GC run and the data has been downloaded to the PC, use the "Batch" and the "Disk Reprocessing" programs to optimize the chromatographic analyses. These programs allow the analyst to change method parameters to improve peak detection and quantitation.
- 10.7.2.8. Create a Peak summary file by using the Peak Summary software. The Peak Summary software summarizes the data from the GC run for the analyst to review. It also creates a .PRN file for input into LOTUS.
  - 10.7.2.8.1. Include the Blank and all samples in the .PRN File.
  - 10.7.2.8.2. If samples from different media (e.g. water and sediment) are part of the same GC run, make a separate .PRN file for each type of sample.
  - 10.7.2.8.3. Store the .PRN file in the S:drive.
- 10.7.2.9. Enter the LOTUS software and input the .PRN file into LOTUS. In LOTUS the data from the GC run is put into a format that can be downloaded to the "VAX."

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10.7.2.9.1. Call up the MACRO "ALT A" and follow the menu.

10.7.2.9.2. Edit the MQL table.

10.7.2.9.3. Create a worksheet file (.WK1) with the final data and store in the S: drive. The file name should include the analyst initial, GC run designation, and the month and year (e.g. RMC 6-90.WK1). R is the initial of analyst, MC is the GC run designation, and 6-90 is the date, June 1990. Print a copy of this file, write the date the file was created, the signature of the analyst and the name of the file on the hardcopy and place it in the project file.

10.7.2.9.4. Create a .VAX file from the .WK1 file. Be sure the name of the file begins with GC with the month and day it will be sent. Also, include the designated initial of the analyst (e.g. S:GCRMC615.VAX). The R is the designated initial, MC is the GC run designation, and 615 is the date, June 15. NOTE: This filename can be no more than 8 characters plus the .VAX extension. Store this file in the S:Drive. Make a hardcopy of the file and write the date, signature of the analyst, and the name of the file and place the hardcopy in the project file.

10.7.2.9.5. The XTALK software program is used to transmit the GCXXXXXX.VAX file to the "VAX" for storage. At the DOS prompt enter the GCVAXFER procedure. This procedure sends a file in the S:Drive that starts with the characters GC with an extension of .VAX (e.g. GCRMC615.VAX) to the Microvax computer. Make sure only one file in the S:Drive starts with GC with the extension of .VAX when you invoke the GCVAXFER procedure. This procedure also processes the file on the Microvax and moves it from the S: drive to the R:Drive on the PC.

10.7.2.10. Copy the method (.MET), sequence (.SEQ), peak summary (.PRN), worksheet (.WK1), "VAX" (.VAX), and raw data files (.HDR, .PTS, and .ATB) created by and for a GC run to the drive designated for the analyst. In most cases the raw data files will already be in this drive.

10.7.2.11. While in the drive designated for the analyst, enter the PKARC...M program. This program archives all files by "crunching" them into one file with an extension of .ARC. This process decreases the storage capacity required by 40 to 50%

10.7.2.11.1. Make sure all files needed to recreate the GC run and data process are archived together.

10.7.2.11.2. Create an archive file name (e.g. RMC6-90.ARC). This file should include the initial designated for the analyst (e.g. R), GC run designation (e.g. MC), and the month and year of the GC run (e.g. 6-90) with the extension.ARC.

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10.7.2.12. Copy the archive file to the Syquest disk under the subdirectory designated for the analyst. The Syquest disk is used to store all archive files and/or files created by and for a GC run and data processing.

- 10.7.2.13. After checking to make sure all files for a project has been stored and can be retrieved, then delete these files from all disks except the Syquest disk.
- 10.7.2.14. All hardcopies of the GC run and data processing should be stored in the Project file.

#### 10.7.3. Dilutions

- 10.7.3.1. The GC chemist is responsible for verifying that all sample extract vials were received from the extraction lab. The chemist is responsible for the vials until GC analysis is complete, and the vials have been stored in proper order or have been discarded.
- 10.7.3.2. Re-mark all vials at the meniscus after dilutions or GC analysis. Do not allow original vials to remain in auto-samplers over the weekend.
- 10.7.3.3. Record whether the dilutions are auto or manual in the GC Logbook.
- 10.7.3.4. Samples requiring dilutions will be analyzed in duplicate one from every set extracted. Select the sample requiring the greatest dilution. If the original dilution was made using the auto-diluter, then its duplicate should be diluted manually or by a different auto-diluter.
- 10.7.3.5. The duplicate dilution shall be made and analyzed as soon as possible after the initial dilutions are analyzed.
- 10.7.3.6. Vial labels and GC logbooks shall indicate which dilution is the duplicate dilution.
- 10.7.3.7. Data from duplicates of the greatest dilution containing usable peak(s) shall agree within ten percent RSD.
- 10.7.3.8. If results are unsatisfactory, resolve the problem before continuing by:
  - 10.7.3.8.1. Rediluting the sample extract in question.
  - 10.7.3.8.2. If the result still exceeds 10% RSD, redilute two additional samples to clarify the validity of the data.
  - 10.7.3.8.3. If unsatisfactory results are obtained, then all samples shall be rediluted from the original extract and

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analyzed by an analyst designated by the chemist in charge of GC analysis.

10.7.3.9. Manual dilutions shall be prepared as follows:

Dilution Desired	_	ume Micro- ringe-Size)	Solvent Volume Microliters	
2%	300	(500)	300	
3 <b>x</b>	200	(250 or 500)	400	
4 <b>X</b>	150	(250)	450	
5 <b>X</b>	200	(250 or 500)	800	
10 <b>x</b>	100	(100)	900	
20 <b>x</b>	50	(50 or 100)	950	
25 <b>x</b>	40	(50 or 100)	960	
40X	25	(25 or 50)	975	
50 <b>x</b>	20	(25 or 50)	980	
100x	10	(10 or 25)	990	
1,000x	20% of	50X		
10,000x	100X o	f 100x	<b></b>	
00,000x	10x of	10,000x		

10.7.3.10. The auto-diluter must be rinsed at least 5 times when diluting sample extracts known or suspected of containing high compound concentrations. Rinsing 3 times is satisfactory for routine samples.

#### 10.7.4. Labeling Chromatograms

- 10.7.4.1. The outside sheet will contain the following information: book number and page, project name, who calculated the data, who checked the calculation, and when and who recorded the data and QC. Use black ink for all labeling and calculations.
- 10.7.4.2. Individual chromatograms will contain the following information: sample number, volume and dilutions, calculation of concentrations, a check mark by each concentration to indicate it was checked, and a yellow highlight on any value that is recorded on the data sheet.
- 10.7.4.3. Do not use "Liquid Paper" to correct any errors. Put one line through the error with initials and date.

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#### 10.7.5. Retention Time (RT) Windows

#### 10.7.5.1. Retention time window size

10.7.5.1.1. Make three injections of all single component mixtures, multiresponse pesticides and PCBs throughout the course of a 72-hour period.

10.7.5.1.2. Calculate the standard deviation of three absolute retention times for each single component pesticide. For multiresponse pesticides/PCBs, choose one major peak from the envelope and calculate the standard deviation of the retention time of that peak.

10.7.5.1.3. Three times the standard deviation of the retention time for each pesticide/PCB will be used to establish the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multiresponse pesticide/PCBs, the analyst should utilize the retention time window but should primarily rely on pattern recognition. If the standard deviation of any compound near the same retention time.

10.7.5.1.4. The laboratory must calculate retention time windows for each pesticide/PCB on each GC column used at the beginning of the program and whenever a new GC column is installed.

#### 10.7.5.2. Daily retention time windows

10.7.5.2.1. Inject all individual standard mixes and all multiresponse pesticides/PCBs at the beginning of each run. To establish the RT window for the pesticides/PCBs of interest, use the absolute RT from the above chromatograms as the midpoint, and  $\pm$  three times the standard deviation calculated in Section 10.7.5.1 as the range.

10.7.5.2.2. Intersperse a standard mixture after every 5 samples to verify that standard retention times are falling within the windows. Any pesticide outside of its established time window requires immediate investigation and correction before continuing the analysis. Reinject all samples following the last standard meeting the criteria. If no peaks are present in the samples, and the surrogate recovery is within limits, no reinjection is necessary and MDLs may be calculated.

#### 10.7.6. Calibration

10.7.6.1. The gas chromatographic system should be calibrated using the external standard technique for all columns used for quantitation.

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10.7.6.1.1. Prepare calibration standards at a minimum of three concentration levels (preferably five) for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with iso-octane or an appropriate solvent. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should define the working range of the detector. This should be done on each quantitation column and each instrument at the beginning and each time a new column is installed.

10.7.6.1.2. Using injections of 1 to 5 uL of each calibration standard, tabulate peak height or area responses against amount injected. The results can be used to prepare a calibration curve for each compound.

#### 10.7.7. Daily GC Column Performance Criteria

10.7.7.1. For packed GC column, saturate or prime the column with a standard mixture containing DDT or PCB's. Recommended concentration is approximately 20 times higher than the quantitation standard. Adjust the carrier flow rate and oven temperature so that 4,4'-DDT has a retention time of approximately 12 minutes for packed columns.

10.7.7.2. Inject a GC/EC column performance mix for packed and capillary columns consisting of:

	<u>nq/uL</u>
aldrin	0.005
endrin	0.025
p,p'-DDT	0.06

at the beginning of each run and after every tenth sample. DDT and endrin breakdown shall not exceed 20% for each. See suggested maintenance in Section 10.6.12 if degradation exceeds 20%.

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#### % breakdown for Endrin =

Total Endrin degradation peak areas (Endrin Aldehyde + Endrin Ketone) $_{x100}$ Total Endrin Peak Area (Endrin + Endrin Aldehyde + Endrin Ketone)

10.7.7.3. All calculations for % breakdown must be on the performance mix chromatogram.

10.7.7.4. Linearity is checked at the beginning of each new project or GC run by injecting three (preferably five) concentration levels of a standard mixture. If the run is for confirmation (no quantitation) or for MDL's, the linearity check is not required. For MDL's, however, a calibration standard at or near the MDL level is required. The following mixture is included as an example:

	Concentrationng/uL				
lindane	0.001	0.002	0.004	0.008	0.016
heptachlor	0.002	0.004	0.008	0.016	0.032
aldrin heptachlor	0.002	0.004	0.008	0.016	0.032
epoxide	0.002	0.004	0.008	0.016	0.032
endosulfan I	0.002	0.004	0.008	0.016	0.032
dieldrin	0.002	0.004	0.008	0.016	0.032
endrin	0.002	0.004	0.008	0.016	0.032
endosulfan II	0.004	0.008	0.016	0.032	0.064
p,p' DDT	0.005	0.01	0.02	0.04	0.08
methoxychlor	0.02	0.04	0.08	0.16	0.32

10.7.8. The % RSD is calculated on representative compounds of interest (e.g. lindane, endrin, p,p' DDT, and methoxychlor). If the % RSD is  $\leq$  20% RSD ( $\leq$  10%, drinking water) for the representative compounds, all compounds are assumed to be linear. Calculate the % RSD for the representative compounds as follows:

Determine the response factor for each concentration by dividing the area or peak height by the amount injected. Calculate the standard deviations of the 5 response factors using:

$$\mathbf{s} = \frac{1}{|\mathbf{n} \times \mathbf{x}^2 - (\mathbf{x} \times \mathbf{x})^2|}$$

and then & RSD:

^{*} Relative Standard Deviation = Standard Deviation x 100

^{*} RSD may also be calculated using the S factor table.

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10.7.8.1. If the linearity criteria is exceeded, see Section 10.6.12 for suggested maintenance.

- 10.7.8.2. The tRSD may be calculated using 3, 4, or 5 concentration levels. However, any peaks quantitated must fall within the selected concentration range, and, the required minimum quantitation limits must be met.
- 10.7.8.3. The calculation for % RSD for the representative compounds must be included in the chromatogram package.
- 10.7.9. Check for peak tailing. The maintenance outlined in 10.7.12 is necessary if peak tailing is noted.
- 10.7.10. Check retention times and compound response compared to previous day or to the beginning of run. If significantly different, check the GC for problem (i.e. septum and/or column leaks, bad syringe).
- 10.7.11. Intersperse standards after every fifth sample. The calibration factor of a specific standard compound shall not exceed a 20% difference from the initial response when screening samples or more than  $\pm$  15% for any standard used for quantitating.

*For multi-response pesticides/PCBs use the response of the major peaks used for quantitation.

Percent difference= 
$$\frac{R_1 - R_2}{R_1}$$
 x100

Where  $R_1$  = Calibration Factor from first analysis  $R_2$  = Calibration Factor from succeeding analysis

10.7.11.0.1. All calculations for calibration factor % difference must be on each standard chromatogram.

#### 10.7.12. Suggested Maintenance

- 10.7.12.1. Corrective measures may require any one or more of the following remedial actions:
  - 10.7.12.1.1. Packed columns For instruments with off-column injection; replace the demister trap, clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert. Inspect the injection end of the column and remove any foreign material (broken glass from the rim of the column or pieces of septa).
  - 10.7.12.1.2. Replace the glass wool with fresh deactivated glass wool. Also, it may be necessary to remove the first few millimeters of packing material if any discoloration is noted,

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10.7.12.1.2. Replace the glass wool with fresh deactivated glass wool. Also, it may be necessary to remove the first few millimeters of packing material if any discoloration is noted, also swab out the inside walls of the column if any residue is noted. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body (described below) and/or repack/replace the column.

10.7.12.1.3. Capillary columns - Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert. Break off the first few inches, up to one foot, of the injection port side of the column. Remove the column and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the column.

10.7.12.1.4. Metal Injector Body - Turn off the oven and injection port heaters and remove the analytical column when oven has cooled. Remove the glass injection port insert (instruments with off-column injection or Grob). Inspect the injection port and remove any noticeable foreign material.

10.7.12.1.5. Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone, toluene and then iso octane, catching the rinsate in the beaker.

10.7.12.1.6. Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol, acetone and hexane. Reassemble the injector and replace the GC column.

#### 10.7.13. Qualitative Analysis

10.7.13.1. Identification of compounds by retention times must be performed by experienced gas chromatographers because slight shifts in retention times require judgment decisions. Observe retention time shifts of standards throughout a days run to evaluate retention time shifts in samples. Utilize the daily retention time windows (See Section 10.6.5.2) for compound identification.

10.7.13.2. Confirm all compounds on a second different column, or different detector (other than FID), unless the compound has been confirmed by GC/MS.

10.7.13.3. At least one sample from a set should be confirmed by GC/MS, if concentration permits. It is the responsibility of the GC analyst to report any pesticides/PCBs confirmed by GC/MS. This must be properly noted on the data sheet. Alpha-BHC, gamma-BHC, endosulfan I and II and endrin must be confirmed on the pesticide

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extract from water rather than the BNA extract (these compounds are unstable at the basic pH).

10.7.13.4. Reporting Chlordane - Weathering and/or different formulations of chlordane may modify the technical chlordane pattern. If the chlordane pattern in a sample is similar to technical chlordane, use a technical chlordane standard for quantitation. (Similar means: all constituents are present, including heptachlor, in about the same ratio as a standard of technical chlordane.) If the pattern is different but gamma and alpha chlordane and other chlordane constituents are present, use the chlordane constituent standards for calculation. Report the individual constituents on the data reporting sheet. Report a total of all constituents listed on the data sheet, except heptachlor, when reporting fish data or when total is requested. Heptachlor is reported separately in these situations.

### 10.7.13.5. Calculation and Project Wrap-up (Also see Section 10.11 on Data Reporting

10.7.13.5.1. To simplify the checking of calculations, everyone must use the same formula for calculating concentrations:

uL injected

X (mL, mg or gm extracted) = Y
(Volume extract in uL) (dilution)

Peak height or area of sample X nq of standard

Peak height or area of standard Y = Concentration

- 10.7.13.5.2. Calculation of Off-scale Peaks Using Area is allowable if it has been shown that response is linear in the concentration range of the off-scale peak and no interfering or rising baseline exists.
- 10.7.13.5.3. All calculations must be checked by someone other than the person who performed the original calculation. Put a check mark after each concentration to indicate it was checked. The chromatogram with the appropriate standards and QC showing the calculations for the reported data should be given to the checker. A hardcopy of the chromatogram should be put in the project file.
- 10.7.13.5.4. The checker should check for accuracy of the transcription of data to the data sheets and the computer "VAX" file.
- 10.7.13.5.5. Diluted samples and all standards should be discarded at the completion of each project to avoid unnecessarily cluttering up the lab and boards.

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10.7.13.5.6. All vials that are ready for disposal should be placed in the "Oily Waste Safety Can." These vials must be treated as hazardous wastesand disposed of accordingly (See Section 4.6).

10.7.13.5.7. All original sample vials are placed in boxes and stored in a locked custody room or locked refrigerator after completion of analysis. See Section 10.8 for instructions on vial storage.

10.7.13.5.8. The project chemist is responsible for calculating surrogate and matrix spike recoveries and recording the results on the appropriate computer data sheet. Unusual results on QC data should be reported to the Pesticides Senior Staff Specialist.

10.7.13.5.9. Samples having greater than or equal to 50 PPM PCBs should be reported to the Extraction Senior Staff Specialist.

#### 10.8. GC/MS ANALYSIS

#### 10.8.1. GC Screen and GC/MS Logbook

10.8.1.1. Record all pertinent information requested on the logbook sheet. See Forms 10-4 and 10-5.

10.8.1.2. Record file name under sample number column as it exists on the disk.

#### 10.8.2. GC Screen

#### 10.8.2.1. Volatile Organics

10.8.2.1.1. All samples should be screened by GC/PID/HALL to determine the approximate concentration level prior to GC/MS analysis. Dilutions for GC/MS analysis are to be determined from this screen analysis.

#### 10.8.2.2. Semivolatile Organics

10.8.2.2.1. All samples should be screened by GC/FID, GC/HALL, GC/FID, or any combination of these necessary to determine the approximate concentration level prior to GC/MS analysis. Dilutions for GC/MS analysis are to be determined from this screen analysis.

#### 10.8.3. File Name Labeling

10.8.3.1. Use the following format for file names for volatile blanks and standards.

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- 10.8.3.1.1. 40VB040885 First two digits are instrument designations, followed by VB for blanks or VS for standards, followed by date of analysis.
- 10.8.3.2. Use the following format for file names for semi-volatile blanks.
  - 10.8.3.2.1. <u>Bl949SLW</u> B for blank, followed by ASB log number for first sample in the set that blank applies to, followed by appropriate analysis designations.
- 10.8.3.3. Use the following format for file names for semi-volatile standards.
  - 10.8.3.3.1. <u>S1949SLW04</u> Surrogate standard. S for standard, followed by ASB log number for first sample in the set that standard applies to, followed by appropriate analysis designations, followed by the day of the month.
  - 10.8.3.3.2. <u>41BA040885</u> First two digits are instrument designations, followed by an abbreviated designation for standard name, followed by date of analysis.
- 10.8.3.4. Sample file name. Use the ASB log number followed by the proper analytical descriptor (ie 42361SLW). See Table 10-1.
- 10.8.3.5. Current instrument designations are:
  - 40 4000 instrument (oldest 4023 system)
  - 41 4100 instrument (newest 4023 system)
  - 51 5100 instrument
  - 50 INCOS 50 EPA
  - 52 INCOS 50 ESAT
- 10.8.3.6. Add the following designations between the SAD number and the analytical descriptor (ie. 40849XDSLS):
  - 10.8.3.6.1. X and Y for duplicates.
  - 10.8.3.6.2. D Dilution (Indicate D2, D3, etc. for subsequent dilutions)
  - 10.8.3.6.3. R, RS, R3, etc. Designates a re-extraction of a sample or reinjection or a purging of a replicate VOA sample.
  - 10.8.3.6.4. If other designations are needed, record their meaning in logbook.
- 10.8.3.7. NOTE: The first eight characters of the file name must be definitive and unique.

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10.8.4.1. File name.

10.8.4.2. Instrument designation.

10.8.4.3. Sample volume information (including dilution information).

10.8.4.4. GC Column type and conditions as 50-210 X 8, I2 F12 where 50-210 is initial and final temperature, X8 is program rate, I2 is initial hold time, F12 is final hold time.

#### 10.8.5. Mass Scale Calibration Using FC43

10.8.5.1. Tune instrument using the following guidance:

10.8.5.1.1. Admit FC43 with carrier flow entering source and at 10⁻⁶ range or as appropriate for the individual instrument.

10.8.5.1.2. Adjust resolution potentiometers to achieve 90-100% resolution across full mass range as nearly as possible.

10.8.5.1.3. Make appropriate tuning adjustments to achieve the following ion intensity ratios as nearly as possible.

Mass 219 15-40% of Mass 69.

Mass 220  $\geq$  Mass of 70.

Mass 414 50-125% of Mass 220.

Mass 131 + 80-120tof Mass 219.

10.8.5.2. Acquire at least 5 scans of FC43 data scanning a mass range of 20-550 amu in profile mode.

10.8.5.3. Run calibration routine.

10.8.5.4. Instrument should calibrate from at least 28 - 502 amu.

#### 10.8.6. Zero the Instrument

10.8.6.1. Set instrument zero consistent with manufacturer's specifications and/or to proven, reliable setting. Acquire 10 scans of data and check the % computer time and peaks/scan parameters. The % computer time should usually be less than 12%. The peaks/scan should usually be less than 100.

#### 10.8.7. Instrument Tuning Performance Test

10.8.7.1. A tune performance check must be performed every 12 hours during analysis.

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10.8.7.2. Analyze 50 ng of Decafluorotriphenylphosphine (DFTPP) for extractables or 50 ng of p-Bromofluorobenzene (BFB) for purgeables.

10.8.7.3. Other concentrations or compounds may be used as required by the analytical protocols.

#### 10.8.7.4. Operating Conditions

10.8.7.4.0.1. Hass Spectrometer parameters same as analysis planned for the day.

10.8.7.4.0.2. GC conditions consistent with prior analysis of reference compound. The reference compound should not elute earlier than 5 minutes from the point of introduction.

- 10.8.7.4.1. The mass spectrum must be acquired in the following manner: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan within 10 scans prior to the beginning of elution of the performance compound.
- 10.8.7.4.2. Compare the ion intensity ratio of those of published criteria (such as those for DFTPP).
- 10.8.7.4.3. If the required criteria are not met, the instrument must be retuned until the spectra meets the specified criteria.
- 10.8.7.4.4. Check retention time and peak shape of reference compound to determine if they are consistent with prior results.
- 10.8.7.4.5. Check the peak intensity (by peak height or area) to determine if the sensitivity is adequate.
- 10.8.7.4.6. Hard-copy a list of masses and intensities and a copy of the chromatogram with areas of each peak printed and maintain in the GC/MS logbook

#### 10.8.8. GC/MS Linearity Check

#### 10.8.8.1. Initial Calibration

10.8.8.1.1. The GC/MS system must be initially calibrated with all compounds of interest at a minimum of three concentrations (5 levels are recommended). Using the response factors (RF) from the initial calibration, calculate the percent relative standard deviations (% RSD) for all compounds.

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10.8.8.1.1.1. A system performance check must be met for all compounds. A minimum response factor of 0.100 for the volatile compounds and 0.05 for the semivolatile compounds is required. If this criteria is not met, corrective action must be taken.

10.8.8.1.1.2. The * RSD for each compound must be less than 20 percent. If this criteria is not met, corrective action must be taken. This might require instrument maintenance, new standards preparation, and/or repeating the analysis of the curve.

10.8.8.1.1.2.1. Thirty percent RSD is acceptable for the following troublesome semivolatile compounds: benzyl alcohol, benzoic acid, 4-nitrophenol, 2,4-dinitrophenol, 2-methyl,4,6-dinitrophenol, 3,3-dichlorobenzidine. If these analytes are of specific importance to the project, corrective action may be necessary. Otherwise, these compounds must be flagged as estimated (J) if the &RSD is greater than 20%.

10.8.8.1.1.2.2. The RF for each compound in each concentration level of the curve must be compared to the average RF of the curve to determine if any individual point on the curve is an outlier. Calculate the percent difference between the average response factor from the curve and the response factor from the individual concentration level in the curve. If the percent difference for any compound is greater than 25%, corrective action may be necessary. This usually means re-analyzing the bad point on the curve.

#### 10.8.8.2. Daily Calibration Check

10.8.8.2.1. A standard mixture containing all volatile or semivolatile compounds of interest must be analyzed every 12 hours of operation.

10.8.8.2.1.1. A system performance check must be met for all compounds. A minimum response factor of 0.100 for the volatile compounds and 0.05 for the semivolatile compounds is required. If this criteria is not met, corrective action must be taken.

10.8.8.2.1.2. A calibration check of the initial calibration curve is made for each target compound. Calculate the percent difference between the average response factor from the initial calibration and the response factor from the current standard. If the percent difference for any compound is greater than 25%, corrective action may be necessary. The analyst must immediately judge the impact on the data generated for that day. Any compounds with %D greater than 25% should be flagged as estimated (J). If more than 25% of the

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compounds are greater than 25%D, corrective action must be taken. This may require generation of a new curve.

RF_I - Average response factor for initial curve

RFC - Response factor from current standard mixture

10.8.8.2.1.3. Continuing without corrective action may be prudent if the outlier compounds are of no particular interest to the project. The Senior Staff Specialist, GC/MS Unit Chief, or Organic Section Chief must be consulted before continuing without corrective action. In this case the corrective action may be to report these compounds as not analyzed or with an estimated flag.

10.8.8.2.1.4. A file of the results from the initial and continuing calibration checks must be maintained. Continuing calibration files are part of the daily standard chromatograms and are to be filed with the appropriate project.

#### 10.8.9. Analyze Standard Mixture

10.8.9.1. Analyze standard mixtures and performance compounds at least every 12 hours.

10.8.9.2. Use GC conditions and MS parameters consistent with sensitivity requirements and equal to those planned for the day's operations.

10.8.9.3. Incorporate internal standards where feasible.

10.8.9.4. Perform system performance check and daily calibration check.

10.8.9.5. Record area count of the quantitation ion for at least one of the internal standards.

10.8.9.6. Analyze surrogate standard appropriate for sample batch.

#### 10.8.10. Analyze Laboratory Blank

10.8.10.1. Utilize internal standards where feasible.

10.8.10.2. Record integrations for the same internal standards recorded in standard.

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10.8.10.2.1. If the area count is not within - 50% to + 100% of those in Standard Mixture, rerun.

10.8.10.2.2. Internal standard retention times must be within + 10 scans or 10 seconds of standard, whichever is greater.

10.8.10.3. Check for carryover from standard injection.

10.8.10.4. Compute surrogate recovery.

#### 10.8.11. Analyze Samples

10.8.11.1. If area count of internal standard is not within - 50% to +100% of the standard, rerun.

10.8.11.2. Internal standard retention times must be within  $\pm$  10 scans or 30 seconds of standard, whichever is greater.

10.8.11.3. Disperse field or lab blanks throughout the day as necessary.

10.8.11.4. Disperse standard mixtures between at least every 12 hours of analysis.

10.8.11.5. Utilize internal standard where feasible.

10.8.11.6. Compute surrogate recovery and record in GC/MS Log.

#### 10.8.12. Setting Up and Analysis of Purgeable Organics

10.8.12.1. Remove standards/surrogates from freezer and let stand at room temperature at least 30 minutes before using.

10.8.12.2. Calibrate MS with FC43.

10.8.12.3. Analyze BFB for Instrument Tuning Performance Test: (make sure filament is off while solvent elutes from column).

10.8.12.3.1. If specs are not met, check for "divots" - severe divots are indications of EM noise, zero high or low, or bad resolution - generally will require you to adjust resolution or recalibrate or rezero.

#### 10.8.12.4. Condition Trap

10.8.12.4.1. Purge blank water onto trap.

10.8.12.4.2. Desorb trap at 180°C on GC column and start normal GC program.

10.8.12.4.3. Bake trap for 15 minutes.

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10.8.12.4.4. Repeat the purge, desorb, and bake cycle from above.

10.8.12.4.5. The analytical sequence should begin with the target standard mixture, followed by a reagent blank, and then samples.

10.8.12.5. Analyze at least one check sample monthly.

10.8.12.6. Potable water samples with positive results should be verified by analyzing a replicate sample whenever possible. The Senior Staff Specialist, GC/MS Unit Chief, or Organic Section Chief should be contacted if deviations from this policy are necessary.

# 10.8.13. Data Processing

10.8.13.1. Plot total ion current profiles.

10.8.13.2. Using the peak finding algorithm in MAP and the total ion current profile, place scan numbers in scan list. The map parameters should be set to find all peaks at approximately 10% of instrument MQL (This is usually set to 10% of the area of an internal standard response.)

10.8.13.3. Print a copy of enhanced spectra and enhanced library search (best 3 match graph, ranked on purity).

10.8.13.3.1. If peaks are asymmetrical, print a spectra with the background manually subtracted.

10.8.13.4. Compare the spectra of the unknown with the 3 best matches and see if one is a logical match.

10.8.13.5. Check for presence of molecular ion and isotopic clusters.

10.8.13.6. Check the purity data printed with the best entries from the library search as an aid to the visual comparison of an unknown spectra to the library spectra.

10.8.13.6.1. Purity of 400, not a good match.

10.8.13.6.2. Purity of 400-600, slight possibility (check fit data; if high, could be two or more co-eluting compounds).

10.8.13.6.3. Purity of 600-800, possibility (also check fit data).

10.8.13.6.4. Purity of 800-1000, high probability of match.

10.8.13.7. If no reasonable match, check 8 Peak Index and other published data bases, as needed.

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## 10.8.14. Qualitative Analysis

10.8.14.1. Target compounds shall be identified by comparison of the sample mass spectrum to the mass spectrum of a standard of a reference spectra of suspected compound. Two criteria must be satisfied to verify the identifications: (1) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

10.8.14.1.1. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within 12 hours of the sample. The RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

10.8.14.1.2. The requirements for qualitative verification by comparison of mass spectra are as follows:

10.8.14.1.2.1. All ions present in the standards mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) <u>must</u> be present in the sample spectrum.

10.8.14.1.2.2. The relative intensities of ions specified above must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

10.8.14.1.2.3. Ions greater than 10% intensity in the <u>sample</u> spectrum but not present in the <u>standard</u> spectrum must be considered and accounted for by the <u>analyst making</u> the comparison. Do not report any compounds with a calculated value below 0.1 of the MQL.

10.8.14.2. A library search shall be executed for Non-Target sample components for the purpose of tentative identification. For this purpose, the most recent available version of the EPA/NIH Mass Spectral Library should be used.

10.8.14.2.1. Do not report any compounds with a calculated value below 0.1 of the MQL. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

10.8.14.2.2. Guidelines for making tentative identification:

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10.8.14.2.2.1. Relative intensities of major ions of the reference spectrum (ions greater than 10% intensity of the most abundant ion) should be present in the sample spectrum.

- 10.8.14.2.2.2. The relative intensities of the major ions should agree within  $\pm$  20%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.
- 10.8.14.2.2.3. Molecular ions present in reference spectrum should be present in sample spectrum.
- 10.8.14.2.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- 10.8.14.2.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination of coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.
- 10.8.14.2.2.6. If in the opinion of the mass spectral specialist, no valid tentative identification can be made, the compound should be reported as <u>unidentified compound</u>. The mass spectral specialist may give additional classification of the unknown compound, if possible (i.e. unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound).
- 10.8.14.2.2.7. Non-Hazardous Substance List compounds identified in samples will be reported using the NBS Library name of the best probable match. The best probable match is selected by the mass spectroscopist from the best matches as chosen by the library search routine ranked by purity. The analysts interpretation may supersede the computer matching algorithm.
- 10.8.14.2.2.8. The NBS Library nomenclature should be stripped of numbers or letters that would make the reported compound a specific isomer (i.e. 1,2-dibromoethane should be reported as dibromoethane).
- 10.8.14.2.2.9. Where more than one isomer of a compound is identified, they should be reported under one name. The total concentration should be reported with this one name and the number of isomers should be reported in parenthesis. The isomer name chosen for one sample of a project should be used in all samples for the project, where no distinguishable spectral differences are present

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(i.e. If the best match for C₃alkyl benzenes is methyl ethyl benzene instead of trimethyl benzene, or propyl benzene, report as methyl ethyl benzene in all samples of the project where this is true).

10.8.14.2.2.10. Name alkyl substituted analogs of Target compound isomers using the earlier eluting of the isomers(i.e. methylfluoranthene, not methylpyrene).

### 10.8.15. Quantitation

10.8.15.1. Target components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte. The EICP area of characteristic ions of analytes are used. The response factor (RF) from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a response factor is calculated using the secondary ion.

10.8.15.1.1. Any compound that had a RRSD in RF of greater than 30 in the initial calibration curve must be reported with an estimated value flag (J). Similarly, any compound that had a t difference in RF of greater than 25 between the RF from daily standard mixture and the average RF from the initial curve must be reported with an estimated value flag (J).

10.8.15.2. An estimated concentration for Non-Target components tentatively identified shall be quantified by comparison to an internal standard free of interferences. The following order of preference for internal standards to use as a reference for extractables is  $\rm D_{10}$ Phenanthrene,  $\rm D_8$ Naphthalene,  $\rm D_{10}$ Acenaphthylene,  $\rm D_{12}$ Chrysene,  $\rm D_{12}$ Perylene, and  $\rm D_4$  Dichlorobensene. Total area counts or peak heights from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A RF of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

### 10.8.16. GC/MS Data Transfer

10.8.16.1. After the data is processed by the GC/MS computer, it is transferred to the IBM personal computer (PC) using terminal emulation and INCOS data transfer. The data is then adjusted taking into account sample dilution, amount purged or extracted, and dry weight, when applicable. Hard-copies are then made and attached to the specific data sheets. After corrections or additions are made to the data based on further analysis of the chromatograms and mass spectra, the final data product is transferred to the VAX computer.

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10.8.16.1.1. To process the data run on the GC/MS, a namelist is created using the first two digits of the instrument, "NL", and the date of the run (eg.,40NL0711). This namelist (.NL) contains all the samples, blanks and standards run on the GC/MS.

10.8.16.1.2. After GC/MS processing is completed, the NLYFER procedure is then used to merge all the individual sample quantitation files in the above namelist to generate a daily combined quantitation list file (eg., 40NL0711.QN). This namelist would contain all pertinent files to be transferred to the PC.

10.8.16.1.3. The INCOS Data Transfer program transfers this namelist (.NL) and its combined quantitation data file (.QN) from the GC/MS terminal to the VAX-linked PC. This transfer creates the PC namelist (.NAM) and mass spec quan (.MSQ) files (eg.,40%L0711.MAM and 40%L0711.MSQ) on the remote PC.

10.8.16.1.4. The .MSQ quantitation list data is then written into the Formaster data base using the Data Entry program. At this point, the data multipliers are entered into the database to account for sample percent moisture, weight/volume, and dilutions.

10.8.16.1.5. The VOAPRN or BNAPRN procedure is then used to read the respective data from the data base, create print files(.PRN), and hardcopy data reports for each sample.

10.8.16.1.6. After the mass spectra and quantitation lists for the samples are reviewed, any changes or remarks are then made in the .PRN files using the QEDIT program which is accessed by the QVOA or QBNA procedure.

10.8.16.1.7. When no more alterations are necessary, the final data can be copied to the MicroVAI using the procedure VOAXFER or BNAXFER.

# 10.8.17. Copying Data to Nine-Track Mag Tape (INCOS)

10.8.17.1. All samples and standards must be copied to nine-track mag tapes using the EPA program.

10.8.17.1.1. The mag tape should be assigned a number (Calendar year, Instrument #, 4 digit number of tape), (ie. 9050-0056). The tape should be labeled as the following example: EPA, Region IV Mag Tape # 9050-0056.

10.8.17.1.2. After analyses are copied, they should be recorded in the mag tape logbook.

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10.8.18.1. The GC/MS chemist is responsible for verifying that all sample extract vials were received from the extraction lab or GC analyst. The chemist is then responsible for the vials until GC/MS analysis is complete, and the vials have been stored or have been discarded. The extract vials should be stored in the refrigerator designated for semivolatile extracts when not in use.

- 10.8.18.2. Recap all vials that are to be retained as soon as possible after puncturing the septum. Remark the volume on the vial label after injection or dilution.
- 10.8.18.3. Diluted samples and standards should be discarded immediately following injection to avoid unnecessarily cluttering up the lab and extract boards.
- 10.8.18.4. All vials that are ready for disposal should be placed in the "Oily Waste Safety Can." Vials must be disposed of according to the procedures outlined in Section 4.6. Dispose of standard and sample vials containing PCB's and other listed compounds in a separate waste container.
- 10.8.18.5. All original sample vials are placed in boxes and are to be stored in a locked custody room upon completion of analysis. See Section 10.9 for instructions on vial storage.
- 10.8.18.6. The project chemist is responsible for calculating surrogate and matrix spike recoveries and recording the results on the appropriate computer data sheet. Unusual results on QC data should be reported to the volatile or semivolatile Senior Staff Specialist and/or the GC/MS Unit Chief.
- 10.8.18.7. GC/MS Files Chromatograms should be filed numerically according to sample numbers. Files should be labeled with the series of sample numbers on first line. Project name(s) should be listed under this. Chromatograms and each file should be arranged as follows:
  - 10.8.18.7.1. Extraction sheets and data sheets.
  - 10.8.18.7.2. Standards analyzed in order of date run.
  - 10-8-18.7.3. Blanks analyzed in order of date run or sample # of blank series.
  - 10.8.18.7.4. Samples analyzed in numerical order.
  - 10.8.18.7.5. Pertinent GC screening chromatograms.
- 10.8.18.8. Any pesticides/PCBs confirmed by GC/MS must be reported to the Pesticides Senior Staff Specialist to be noted on the pesticide/PCB data sheet. Chromatograms from Pesticide and PCB confirmation are sent to the GC unit to file with their chromatograms.

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10.8.18.9. Keep GC screen chromatograms for samples that did not require GC/MS analysis. All other GC screen chromatograms should be discarded.

### 10.9. Extract Storage

10.9.1. Sample extracts are to be stored in storage containers after final reporting of data. These containers will be kept in their respective areas of the GC Lab and the GC/MS Lab in a refrigerator, based on the type of sample. As soon as possible, the containers should be transferred to the permanent storage area in the middle section of the Hazardous Materials Storage Building. The large refrigerator will be used for this storage. After placing samples in the refrigerator, a custody seal must be placed on the refrigerator door.

# 10.10. Preparation, Storage, and Use of Organic Analytical Standards

#### 10.10.1. Standard Sources

10.10.1.1. Primary Standards: ETD, HERL, US EPA, RTP, NC 27711 Telephone 629-3951; U.S. Food & Drug Administration 202/245-1406; and various commercial sources when unavailable from the first two sources, requesting purest grade available.

- 10.10.1.1.1. Prepared standards; EMSL Cincinnati
- 10.10.1.1.2. QC Standards, EMSL Cincinnati
- 10.10.1.1.3. Prepared standards; commercial. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. If the purity of these standards is questionable, report the data based on these standards as estimated.

# 10.10.2. Glassware, Equipment, and Solvents:

- 10.10.2.1. Analytical balance, capable of an accuracy of ± 0.1 mg.
- 10.10.2.2. Spatula, stainless steel.
- 10.10.2.3. Transfer class " $\lambda$ " pipets and pastuer disposable pipets or suitable syringes.
- 10.10.2.4. Flasks, volumetric, 25, 50, 100 and 200 mL.
- 10.10.2.5. Bottles, Teflon-faced caps 60 mL.
- 10.10.2.6. Small glass funnels, and bent paper clip.
- 10.10.2.7. Refrigerator, explosion proof.
- 10.10.2.8. Pesticide grade solvents: ethyl acetate, toluene, acetone, isooctane, hexane, methanol, and carbon disulfide.

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# 10.10.3. Safety Precautions and Operating Procedures

- 10.10.3.1. Rubber gloves should be used when handling reference materials.
- 10.10.3.2. Standards used for quantitating samples are to be made up by a chemist.
- 10.10.3.3. Hoods should be used when weighing toxic standards or diluting with organic solvents.
- 10.10.3.4. Rinse all glassware prior to use with methanol, acetone, and hexane and let air dry in hood.
- 10.10.3.5. Always perform a balance check with Class-S weights each day the balance is used. Record the balance check on the Standard Sheet. Check calculations on solutions to be made up.
- 10.10.3.6. Do not store any standards in volumetric glassware. Transfer to a 60-mL screwcap bottle with Teflon liner if the solution is to be stored. Use phosphate tubes or vials, with Teflon liners, for short term storage. All standards must be properly labeled.
- 10.10.3.7. Always rinse used glassware with acetone before washing in dishwasher with other glassware. Rinse pipets out with acetone immediately after use.
- 10.10.3.8. Keep all standards in refrigerator when not in use.
- 10.10.3.9. Always let standards and solutions come to room temperature before opening.
- 10.10.3.10. Check new working standard against old standard. Old standard may be slightly more concentrated due to evaporation of solvent from repeated openings.
- 10.10.3.11. Transfer waste standards to a waste bottle. Rinse the empty bottle several times with acetone. Add the rinsate to the waste bottle and discard the standard bottle.
- 10.10.3.12. Provide a large waste beaker located in a hood for rinsing all used glassware and pipets before washing with soap and water. Transfer the wash solvent to a waste bottle.
- 10.10.3.13. Volumetric flasks and storage bottles used for standards must be rinsed several times with distilled water to remove any alkaline residue. Alkaline residues cause degradation of certain organics and pesticides.

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10.10.4.1. Extractable organic and pesticides Orange Mix Stock Standard. Replace every year. Concentration of 1000 ng/uL.

# 10.10.4.1.1. Preparation of stock standards

10.10.4.1.1.1. Weigh 50.0 mg of primary standard into a 50 mL beaker using a small spatula for solids or a disposable pasteur pipet for liquids.

10.10.4.1.1.2. Approximately 2 mL of ethyl acetate is suggested to dissolve standard. Some standards may require placing the beaker in an ultrasonic cleaning device or on a steam bath for complete dissolution. Other choices of solvents to use for dissolving the standard are toluene, acetone, methylene chloride, and methanol.

10.10.4.1.1.3. Transfer through a glass funnel into a 50 mL volumetric flask, washing with an appropriate solvent. Dilute to volume with the least volatile of the appropriate solvents and mix. Calculate the concentration in micrograms per microliter. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. If the purity is less than 96%, the weight must be adjusted for purity.

10.10.4.1.1.4. Transfer to 60 mL screw capped bottles with orange labels. Old bottles that contain the same standard may be reused if rinsed with isooctane.

10.10.4.1.2. <u>Calibration Standards</u> - This consists of a set of five standards with concentrations covering the linearity range for each detector used for quantitative analysis.

10.10.4.1.3. Intermediate and working standards, (blue, yellow, silver, and red) are diluted with a high boiling solvent such as isocotane. Working standards are diluted to give even numbered concentrations if possible, from intermediate and working standards (i.e. 10 ng/uL vs 11 ng/uL). Discard working standards after six months. Some standards are unstable and must be made up more frequently. Working solutions should be checked at least quarterly against available Cincinnati QC samples or a check standard. New working solutions should be checked against the old standard. Percent difference must not exceed 10% for each compound checked.

Percent Difference = 
$$\frac{R_1 - R_2}{R_2}$$
 x 100

Total area of peak*
Amount injected (in nanograms)

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10.10.4.1.4. For multicomponent pesticides/PCB's, use the total area of all peaks used for quantitation.

R₁ = relative response from working standard

Mixes Used	<u>Detector</u>	Concentrations (nq/uL)
Blue	FID/MS	5 - 100
Yellow	Hall, N/P	0.2 - 10
Silver	Hall	0.05 - 2.5
Red	EC	0.005 - 0.25
Green	(Spike solutions	for all parameters)

10.10.4.1.4.1. All standards must be stored in refrigerator when not in use.

10.10.4.2. Spike Solution (green label) - All spike solutions are made from stock or intermediate solutions and diluted with acetone or methanol.

10.10.4.3. <u>Purgeable standard solutions</u> - Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

10.10.4.3.1. Place about 9.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

10.10.4.3.2. Add the assayed reference materials as described below:

10.10.4.3.2.1. Liquids - Using a 100 uL syringe, immediately add 2 or more drops of assayed reference material to the flask, then re-weigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

10.10.4.3.2.2. Gases - Introduced from lecture bottle. Flow rate is controlled with a valve through a teflon tube to top of the meniscus.

10.10.4.3.3. Re-weigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter. When compound purity is assayed to be 96% or greater, the weight may be used

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without correction to calculate the concentration of the stock standard.

10.10.4.3.4. Transfer the stock standard solution into a Teflonsealed screw-cap bottle. Store, with minimal head-space, at  $-10^{\circ}$ C to  $-20^{\circ}$ C and protect from light.

10.10.4.3.5. When stored under the above conditions, those standards should be replaced if comparison with QC check samples indicates a problem. Gases are replaced after 3 months and all others after 6 months.

10.10.4.3.6. Intermediate standards - Using stock standard solutions solutions, prepare intermediate standards in methanol that contain the compounds of interest, either singly or mixed together. The intermediate standards should be prepared at concentrations such that the aqueous calibration standards will bracket the working range of analytical system. Intermediate standards should be diluted to give calibration standards of approximately 30 ng/uL.

10.10.4.3.7. Intermediate standards are prepared weekly and should be stored in a freezer with minimal headspace. They should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards, will be available from the U. S. Environmental Honitoring and Support Laboratory, in Cincinnati, Ohio.

10.10.4.3.8. Internal/Surrogate standard spiking solution — The solution may be prepared as described above. Combine the individual components so that their concentration is equal to 30 ng/uL. 20 uL of this solution is added to 20 mL of a sample; 5 mL of this sample is purged with give a concentration of 30 ug/L (ppb) of each surrogate compound. The intermediate surrogate solution is prepared weekly, or sooner if exhausted through a large number of analyses. The stock standard is good for a minimum of 6 months. The first component is a good barometer of the solution; if it appears to be much smaller in size than the other two in a GC/MS run, the stock solution should be discarded and another one made.

### 10.10.5. Records

10.10.5.1. Stock Standards. Enter the weight of the primary standard and other requested information on the stock standard sheet in the orange section of the "Organic and Pesticide Standard Logbook" (See Form 10-6). Also, record the requested information on the orange card in the file, the summary log sheet at the beginning of the section (See Form 10-12), and on the label of the standard bottle (See Form 10-7). Insure that the corresponding solution number is circled on all three items each time a new stock solution is made up.

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10.10.5.3. Blue, Yellow, Silver, and Red Mixes. Record the parent color, dilution number and date of preparation on the standard sheet (See Form 10-8, 9 and 10). Enter requested information on the color card, the summary logsheet at the beginning of the section and standard bottle (See Form 10-7). Insure that the correct dilution number matches on all items.

10.10.5.4. Keep each section of the Standards Book in alphabetical order.

10.10.5.5. A new standard sheet and color card must be prepared when one or more ingredients or the concentrations in a mixture changes. On the new sheet and color card, circle the next number in sequence from the old sheet (See Form 10-10). Hark the old sheet "Cancelled" with the end of the standard logbook. Discard old color card. Retain the old mix bottle number on the new mix. Select a new number at the end of the sequence when a completely new mix or standard is made up. Retire the number when a mix or standard is no longer needed.

10.10.5.6. In GC and GC/MS Logbook, enter name of the standard plus the color code if applicable.

10.10.5.7. There are three organic pesticide standards logbooks. Volume 1 is for stock solutions (orange label). Volume 2 is for intermediate, working, and spike standards. Volume 3 is for outdated and cancelled sheets.

### 10.11. Data Reporting

#### 10.11.1. General

- 10.11.1.1. No data will be reported until all QC data have been evaluated and data determined to be valid.
- 10.11.1.2. In certain situations, there is a deviation in whether or not % moisture is determined. This deviation should be noted on the data sheet with the proper remark.
- 10.11.1.3. Report parameter concentration in units as in Table
- 10.11.1.4. Waste samples and t moisture Calculate and report waste samples on a dry weight basis if they are primarily heavily contaminated soil or dry solid. Waste that is primarily nonaqueous liquid should be reported using the weight as received or wet weight basis.

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# 10.11.1.5. Use the following designations on the data sheet:

10.11.1.5.1. U - The analyte was analyzed for but not detected. The value preceding the "U" is the "minimum quantitation limit (MQL)".

10.11.1.5.1.1. Minimum Quantitation Limit (MQL) -- Every sample has a concentration level below which the variance of the results for a particular analyte (element or compound) exceeds the acceptable quality control criteria. This level is the MQL and is reported as the value preceding the "U". The MQL is based on the lowest quantitative data point of the instrument calibration curve. The MQL is derived using this data point and other factors such as: sample size, dilution required, sample % moisture, and sample interferences. The value often varies from analyte to analyte within a sample. Analytes are often detected at levels below the MQL and are reported as estimated values (J). Generally, analytes identified below the MQL will only be reported if the concentration is greater than one tenth of the MQL.

10.11.1.5.2. J - The identification of the analyte is acceptable, but the quantitative value is an estimate. The value preceding the "J" is the "estimated value".

10.11.1.5.2.1. Estimated Value—Every sample analysis has quality control criteria associated with the quantitative data which have been established based on similar analyses. When these criteria are exceeded, the value for that analyte or similar analytes is reported as an estimated value. Examples are:

- o calculated values are below or above an appropriate linear range
- o calculated values are below the MQL of an analyte
- o analytical holding times for analysis are exceeded
- o surrogate recovery limits are exceeded
- o there are no known quality control criteria for an analyte.
- 10.11.1.5.3. H There is presumptive evidence that the analyte is present, but it has not been confirmed. The analyte is "tentatively identified".

10.11.1.5.3.1. Tentative Identification—There is an indication that the analyte reported is present. The quality control requirements necessary for confirmation were not met. Examples are:

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10.11.1.5.3.1.1. A specific list of compounds is analyzed for in every organic analysis by gas chromatography/mass spectrometry (GC/MS). Other compounds are often present and their spectra are compared to published mass spectral data. If a qualitative determination is made, the compound is reported as tentatively identified.

10.11.1.5.3.1.2. The presence of analytes is often indicated, but there is evidence of possible interferences. There is presumptive evidence that the analyte is present, therefore, it is reported as tentatively identified.

10.11.1.5.4. C - The analyte is determined to be present. The presence of the analyte was "confirmed by GC/MS".

10.11.1.5.4.1. Confirmed by GC/MS-Pesticides are routinely analyzed by gas chromatography with an electron capture detector (GC/EC). When identified by GC/EC analysis in sufficient concentrations, pesticides are confirmed on the mass spectrometer by comparing the spectra of the analyte with the spectra of a particular pesticide. If a good spectral match is obtained, the pesticide identification is considered to be confirmed. The concentration is quantitated by GC/EC.

10.11.1.5.5.  $\lambda$  - The analyte was analyzed in replicate. The value preceding the " $\lambda$ " is an "average value" of the replicates.

10.11.1.5.5.1. Average Value—Samples are often analyzed in replicate (usually in duplicate). Aliquots of the same sample are analyzed and the values are averaged. Sometimes replicate samples are analyzed and the values are reported as an average.

10.11.1.5.6. K - The analyte is determined to be present. The actual value is known to be "less than" the value preceding the "K".

10.11.1.5.6.1. Less Than Values—The analyte is present, but the amount of the analyte is determined to be below an acceptable level for quantitation. The concentration can not be calculated, but is determined to be less than the value given. Example: 10K means that the analyst has determined that the analyte is present at some undetermined amount less than 10.

10.11.1.5.7. L - The analyte is determined to be present. The actual value is known to be "greater than" the value preceding the "L".

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10.11.1.5.7.1. Greater Than Values—The analyte is present, but the amount of the analyte is determined to be above an acceptable level for quantitation. Example: 500L means that the analyte is present at some undetermined amount greater than 500.

10.11.1.5.8. R - Data is "rejected" and should not be used.

10.11.1.5.8.1. Rejected Data-Some or all of the quality control data for the analyte were outside criteria. The presence or absence of the analyte can not be determined from the data. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.9. UJ - This is a combination of the "U" and "J" codes. The analyte is not present and the value preceding "UJ" is an estimated MQL.

10.11.1.5.10. JH - This is a combination of the "J" and "N" codes. The analyte is tentatively identified and the value preceding the "JN" is estimated.

10.11.1.5.11. JR- This is a combination of the "J" and "R" codes. The analysis indicated the presence of the analyte. The data is rejected and the value preceding "JR" is estimated. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.12. UR - This is a combination of the "U" and "R" codes. The analysis did not indicate the presence of the analyte. The data is rejected and the value preceding "UR" is the MQL. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.13. MAI - Not analyzed due to interference.

10.11.1.5.14. MA - Not analyzed for.

10.11.1.6. The number precedes the code letter in all cases.

10.11.1.7. When adding one or more J values to real values (e.g., for DDTR), J the total if the J values are equal to  $\geq$  10% of the total value.

10.11.1.8. Rules of rounding. Round off by dropping digits that are not significant. If the digit 6,7,8, or 9 is dropped, increase preceding digit by one unit; if the digit 0,1,2,3, or 4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 becomes 2.2 and 2.35 become 2.4.

10.11.1.9. Reporting estimated minimum quantitation limits (MQL) - The MQL is reported to 2 significant figures.

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- 10.11.1.10. Reporting target compounds below the MQL Report the actual calculated value (to 2 significant figures) with a J for any concentration below the MQL. Anything below 1/10 of the MQL and/or less than a 3mm peak height is reported as not detected.
- 10.11.1.11. If dilutions of the sample extract are required, the MOL is raised by the same factor as the dilution.
- 10.11.1.12. Determine from the analytical request if a specific limit of quantitation is required. This is especially important when analyzing samples for compliance monitoring, spill investigations, and drinking water investigations.
- 10.11.1.13. Calculate the MQL for the blank using the <u>lowest</u> sample weight or sample volume.
- 10.11.1.14. Report all non-target (library search compounds) data to 1 significant figure.
- 10.11.1.15. Inorganic compounds. Do not report sulfur,  $E_2S$ ,  $SO_2$ , etc.
- 10.11.1.16. Reporting duplicate data Calculate and report the average with an A flag. If a compound is detected on one duplicate and not the other, do not report the compound.

# 10.11.2. Momenclature for Library Search Compounds

- 10.11.2.1. Spaces in chemical names Be careful where spaces are used in nomenclature especially when rearranging CAS names. Where there are dashes, always attach the words. Where there are spaces, always leave the space.
- 10.11.2.2. Acids. Always precede acids with a space, same for esters, acetates, oxides, etc. (i.e. benzoic acid).
- 10.11.2.3. Isomers and/or rearranged names, 2-Hexanone, 5-methyl-3-methylene -, rearrange and combine to methylmethylenehexanone. Dashes indicate no space when the name is rearranged.
- 10.11.2.4. Alkyls. No space.
- 10.11.2.5. Where spaces are proper, be sure to leave a very pronounced space on the handwritten sheet so it is evident to the data entry person.
- 10.11.2.6. Esters. Do not rearrange esters (i.e., dodecanoic acid, methyl ester not methyl ester of dodecanoic acid).
- 10.11.2.7. Truncated names in data system. The current GC/MS data system library only stores the first 70 characters. Long names with odd looking endings should be looked up to verify the complete name. The HS library display truncates if the name extends beyond where the scan number is printed.

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- 10.11.2.8. Two or more isomers present. Report number of isomers in parenthesis after name (i.e., dimethylbutane (2 isomers)).
- 10.11.2.9. Unidentified compounds. Report as unidentified compounds, and the number of compounds.
- 10.11.2.10. Eydrocarbon series. Report homologous hydrocarbon series as "petroleum product" with an "N" flag in the result field.
- 10.11.2.11. Common names vs CAS names. The following commonly found compounds are changed from CAS names to common names:
  - All pesticides recognized as being pesticides
  - 2-propanone to acetone
  - 2-propanol to isopropanol
  - 2- butanone to methyl ethyl ketone
  - 2-pentanone to methyl propyl ketone
  - 3 methyl-2-butanone to methyl isopropyl ketone
  - 4 methyl-2-pentanone to methyl isobutyl ketone thiobismethane to methyl sulfide

Acetic acid ethyl ester to ethyl acetate

- 1,1'-oxybisethane to ethyl ether
- 2,2'-oxybispropane to isopropyl ether
- 1,2-benzenedicarboxylic acid to phthalic acid ethenylbenzene to styrene

# 10.11.3. Laboratory Contaminants

10.11.3.1. The following organics are frequently detected in blanks in trace concentrations. Therefore, special precautions need to be taken when reporting positive findings of these compounds.

### 10.11.3.1.1. Purquable Organic Analysis

- 10.11.3.1.1.1. Do not report these compounds unless they are 5 times the blank value; benzene, tetrachloroethylene, toluene, ethylbenzene, methylene chloride, trichloroethene, tetrachloroethane, mylenes, and acetone.
- 10.11.3.1.1.2. Isopropanol is used to decontaminate field equipment; therefore, do not report unless a special request is made.
- 10.11.3.1.1.3. Do not subtract blank values from sample values.
- 10.11.3.1.1.4. Sediment contamination by methylene chloride and acetone may be higher than normal water analyses. Therefore, do not report these compounds unless they exceed 10 times the blank value.

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# 10.11.3.1.2. Extractable Organic Analysis

10.11.3.1.2.1. Report phthalates only if above the MQL.

10.11.3.1.2.2. Do not report these compounds unless they are present at 5 times the blank value: bis(2-ethylhexyl) phthalate, diethyl phthalate, dibutyl phthalate, n-nitrosodiphenyllamine, the xylenes, silicones, and phthalic acid.

10.11.3.1.2.3. Butoxyethoxyethanol and related compounds are common contaminants of tubing used in automatic samplers. Therefore, if these compounds are detected in the sampler blank, report them in sampler blank and all samples where identified.

10.11.3.1.2.4. Do not subtract blank values from sample values.

# 10.11.3.2. Reporting of Data when Considering Other Possible Contaminants

10.11.3.2.1. Use the following criteria when evaluating the validity of a positive identification:

10.11.3.2.1.1. If the compound in question is in the blank and in the sample but the concentration in both is <MQL, report the compound as --U.

10.11.3.2.1.2. If a compound in question is detected in the blank at greater than the usual MQL and is in a sample at ≤ the blank, report the sample value as the MQL for the sample.

10.11.3.2.1.3. If the compound in question is in the blank at <MQL but in the sample at >MQL, then use your professional judgement in determining its validity.

10.11.3.2.1.4. In general, it should be >2 times the blank value before reporting. The same is true if the compound in question is present in both the blank and sample, and >MQL.

10.11.3.2.1.5. Natural organics in fish - Do not report cholestanol or related compounds.

10.11.3.2.1.6. Do not report chlorinated and brominated cyclohexenes, cyclohexanes, and cyclohexanols if chlorinated water was extracted with methylene chloride. The person taking the sample should verify that it was chlorinated water and write a memo to the Chemistry Section, so stating.

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10.11.3.2.1.7. Do not subtract blank data from sample values.

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# TABLES

# ORGANIC ANALYSIS

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# ANALYTICAL DESCRIPTORS

Hethod#	Descriptor	Sample Type
46A	VLW	Volatile low water
43D	VLS	Volatile low soil/sed
54B	VMS	Volatile medium soil/sed
54B	VMW	Volatile medium waste
56	VAC	Volatile air canister
47	slw	Semivolatile low water
43	SLG	Semivolatile low soil/sed w/GPC
43A	SLS	Semivolatile low soil/sed wo/GPC
54	SMS	Semivolatile medium soil/sed
54A	SHW	Semivolatile high waste wo/son.
54C	SMW	Semivolatile medium waste w/son.
44	SLT	Semivolatile low tissue
50B	Sap	Semivolatile air PUF
58	SCW	Semivolatile Cartridge ext. water
60	STC	Semivolatiles by TCLP extraction
55	PLW	Pesticide low water
43	PLG	Pesticide low soil/sed w/GPC
43A	PLS	Pesticide low soil/sed wo/GPC
43B	PSA	Pesticide low soil/sed w/acid cleanup
43C	PSH	Pesticide low soil/sed w/hex/acetone
44	PLT	Pesticide low tissue
44 <b>A</b>	PTH	Pesticide low tissue w/hexane
44B	PTA	Pesticide low tissue w/acid cleanup
50B	PAP	Pesticide air PUF
54	_ PMS	Pesticide medium soil/sed
54A	PHW	Pesticide high waste wo/son.
54C	PMW	Pesticide medium waste w/son.
52B	PCW	Pesticide Cartridge ext. water
57	PNP	Pesticide low water nitrogen/phosphorous
60	PTC	Pesticides by TCLP extraction
35	PWO .	PCBs waste oil
38	HLW	Herbicides low water
51	HT.S	Herbicides low soil/sed
60	HTC	Herbicides by TCLP extraction
48	FLW	Formaldehyde low water
48	FLS	Formaldehyde low soil/sed
59	FAC	Formaldehyde air cartridge
61	CLW	Carbamates low water w/HPLC
61	CLS	Carbamates low soil/sed w/HPLC

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# **FORMS**

# ORGANIC ANALYSIS

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SOLUTION NO.	DATE	INT	COMPOUND SOURCE	LOT NO.	PURITY	NET NET	ADJUSTED NET WEIGHT:	SOLVENT	DILUTION VOLUME ml	FINAL CONC. ng/ul	BALANCE CHECK ACTUAL/ FOUND
1.	9/22/82	RRR	RIP	9345	1004	50.0mg	50.0mg	2ml ethyl acetate QB iso- octane	50.0ml	1000	OK
2.	9/22/83	RRR	RTP	9562	100%	50.0mg	SO.Omg		50.0ml	1000	OK
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4.											
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# STOCK COLOR CARD FILE

_6_	Aroclor 1260 1 2 3 4 5 6 7 8 9 10 11	Replace after 1 year						
		ng/ul	DATE	INT				
$\Box$	Aroclor 1260	1000	9/22/81	RRR				
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STOCK BOTTLE LABEL

Code - Aroclor 1260

6 Aroclor 1260 1 2 3 4 5 6 7 8 9 10 11 12 13



# WORKING COLOR CARD FILE

9 Aroclor 1260 1 2 3 4 5 6 7 8 9 10 1	Replace	after 6 mc	nthe
	ng/ul	DATE	INI
1 Aroclor 1260	0.25	9/22/81	RRR
2 -	0.25	3/22/82	RRR
3	0.25	9/22/82	RRR
4	0.25	3/22/83	RRR
5			
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7			
. 8			
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### WORKING BOTTLE LABEL

Code - Aroclor 1260

9 Aroclor 1260 1 2 3 4 5 6 7 8 9 10 11 12 13



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A = Color Code B = Bottle Number C = Dilution Number

# WORKING STANDARDS

6 - Aroclor 1260

	нане			SOLVEN	I: <u>Iso-</u> -	octane			
	DATE	INITIALS			Parent Solution Ident. A -B- C		ALI.	VOL.	FIN. Conc.
_1,1	9/22/81	RRR	Aroclor 1260		Or-6-1	1000	_ 5	50	100
2.	9/23/83	RRR	<u> </u>		Or-6-2	1000	5	50	100
3.									
3. 4. 5. 6. 7.									
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	name			SOLVERT:				
<del></del>	DATE	INITIALS	COMPOUND	Parent Solution Ident. A -B- C	Solution	ALI. VOL.	VOL.	PIN. Conc. ng/ul
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A = Color Code
B = Bottle Number
C = Dilution Number

### WORKING STANDARDS

9 - Aroclor 1260

NAME				SOLVENT:						
	DATE	INITIALS	сонротно	Parent Solution Ident.	Solution ng/ul		VOL.	FIN. Conc. ng/ul		
ļ	9/22/81	RRR	Aroclor 1260	B-6-1	100	5	50	10.0		
2.	3/22/82	RRR	•	B-6-1	100	5	50	10.0		
3.	9/22/82	RRR		8-6-1	100	5		10.0		
4.	3/22/83	RRR	•	B-6-2	100	5	50			
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	NAME			SOLVENT:				
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# Example of ingredient amount change in a mix standard.

λ = Color Code
B = Bottle Number
C = Dilution Number

### WORKING STANDARDS

11 - Consent Degree Solution I

\	NAME			SOLVEN	T:				
,	DATE	INITIADE	сонротно		Parent Solution Ident. A -B- C	Conc.of Parent Solution	ALI.		PIN. Conc.
1.	7/10/79	RH	bindane		Or-11-1		2	100	20
2.	3/22/82	RRR	Reptachlor		0r-49-19	1000	4	100	40
3.	9/22/82	RRR	Aldrin		-0z-1-1	1000	4	100	40
4.	3/22/83	RRR	Bentachlor Roomide	کے	Or-50-1	1000	4	100	40
5.				E	Or-36-1	1000	4	100	40
6.			Dieldrin		Or-35-1	1000	<b>+</b>	100	40
7.			Endrin		Or-39-1	1000	4	100	40
3.			Endosulfan II		02-37-1	1000	8	100	.80
9. 9.			D.D'-80T		Or-28-1	1000	10	100	100
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# 11 - Consent Degree Solution I

	иуна	<del></del> -		SOLVENT:				
	DATE	INITIALS	сонросить	Parent Soluti Ident.	on Parent Solution	ALI.	DIL. VOL.	FIH. Conc.
-14	1/21/81	RRR	Lindane	0r-11	-2 1000	2	100	20.0
-2.			Reptachlor	Or-49	-2 1008	4	100	
_3.			Aldrin	Or-1-	2 1012	4	100	40.5
_4,			Heptachlor Epoxide	0r-50-	-2 1011	4	100	40.4
5.			Endosulfan I	Or-36-	-2 1000	4	100	40.0
6.			Dieldrin	Or-35-	-2 1008	4	100	40.3
5. 6. 7.			Endrin	Or-39-		4	100	40.5
_8.			Endosulfan II	Or-37-	-2 1056	8	100	84.5
9.			D.D'-DDT	OT-28		10	100	
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				C ANALYSIS			
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					l. Y Capilla	1/4 in. OD I	L ID 0.53 I
OC COL	mer TT	P2 :			2. Camilla	Pilm Thickness ry Length Film Thickness	1.5 ···
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B - Bottle Number D - Dilution Number

section			

Name of Standard or Mix	I.B	LD.	Date	1 8	D	Date	1 B	D	Date
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# 11. INORGANIC AMALYSIS, PERFORMANCE QUALITY CONTROL AND AMALYTICAL OPERATION

11.1. Every element of environmental data acquisition, from sample collection to final data reporting, has associated with it degrees of error. The primary purpose of a quality assurance program is the optimization of conditions whereby the introduction of error can be either precluded or substantially reduced. The operating procedures and quality control checks practiced in this laboratory and outlined in this manual are implemented to minimize the total error associated with data generation. No number can be affixed to total error; however, analytical performance is measurable and thus definable. This section is limited to a discussion of the analytical operation and procedures used in this laboratory to measure and record analytical performance.

# 11.2. General

- 11.2.1. During the course of generating data on samples for inorganic parameters, it is be the policy of this Branch to apply the best laboratory practices, use approved methodology when mandated by regulation, use standardized methodology, if possible, when approved methodology is not applicable, fully document all operations associated with the generation of data and to meet certain quality requirements that will be designated in the following paragraphs.
- 11.2.2. Safety precautions associated with the safe handling of toxic chemicals, reagents, solutions and samples will be observed and regarded as a first order responsibility of the analyst. The analyst will take the necessary precautions to prevent exposure or harm both to himself and his fellow workers.
- 11.2.3. Water used to prepare calibration standards, spike solutions, standard reference solutions or any sample dilutions or mixtures must meet or exceed the requirements for Type II grade water as specified by the American Society for Testing and Materials (ASTM). See section 2.2 of Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA 600/4-79-019, March 1979).
- 11.2.4. Reagents must be ACS reagent grade quality or better. All reagents will be dated upon receipt, and will be properly disposed of when the shelf life has been reached.

# 11.3. Custody

11.3.1. All samples removed from the custody room must be signed out. When a sample is signed out, the signee is legally assuming custody of the sample and is responsible for its integrity and accountability during possession. Custody is relinquished only when the samples have been returned and legally signed over to the custody room. Aliquots taken from the original samples for analysis will be accounted for by entering sample ID in the proper log books during preparation and analysis.

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# 11.4.1. Preparation and Analysis

- 11.4.1.1. Drinking Water: Samples (100 mLs ea.) are digested with nitric-hydrochloric (0.5/1.2mLs) acids according to paragraph 9.4 of Method 200.7. The sample is heated on an oscillating hot plate until volume is reduced to 5-10 mLs and made up to 25 mLs for an initial scan by Inductively Coupled Plasma (ICP).
- 11.4.1.2. The concentration factor is limited by the following maximum concentrations: 200 ppm Ca and Na; 100 ppm Mg, Mn, Al & Fe. All positive arsenic and lead values will be confirmed by STPF atomic absorption, methods of additions. Selenium is determined by STPF atomic absorption, methods of additions. Mercury analyses are performed according to MCAWW¹ 245.2.
- 11.4.1.3. Other Waters: Monitoring well, ambient water, effluents and other water samples are digested with nitric-hydrochloric (2.5/5 mLs) acids according to paragraph 9.4 of Method 200.7. All digests are scanned by ICP. Where the detection/quantitation technique is specified by program requirements, positive elements from the ICP scan will be verified by atomic absorption to satisfy the appropriate requirement. Mercury analyses are performed according to MCAWW-245.2.
- 11.4.1.4. Soil and Sediment: A 50 g aliquot is taken from a well mixed sample and weighed in a crucible. The sample is dried overnight 6 60°C for a * moisture determination. The dried sample is ground to fineness and a 1 g subsample is taken for analysis. Sample digestion is conducted according to Method 3050² and made up to 100 mLs for analysis. A scan by ICP (PE6000) provides preliminary data and possible dilution requirements.
- 11.4.1.5. Final low level data for thirty elements is generated by the Jarrell Ash simultaneous instrument. NOTE: Certain samples will contain interferences due to complex matrices or elements not included in the thirty element array of the JA ICP.
- 11.4.1.6. Elements not included in the array cannot be automatically corrected and these samples must be scheduled back to the PE 6000 for graphic examination and proper correction for interferences. Atomic absorption analysis may be required in certain instances.
- 11.4.1.7. A second accepted digestion procedure for soil/sediment samples is contained in "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish." This method is superior to 3050 if silver, antimony and tin are of interest and if the matrix is not high in organic material.

^{1/} MCAWW - Methods for Chemical Analysis of Water and Wastewater. EPA 600/4-79-020, March 1979. (Revised March 1983)

^{2/} SW846 - Test Method for Evaluating Solid Waste, EPA 1982.

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- 11.4.1.8. Hercury analysis of sediments will be conducted according to method 245.5 8.2 in MCAWW.
- 11.4.1.9. Fish: Whole fish are initially prepared with dry ice grinding as per the method in "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue".
- 11.4.1.10. Requests for analysis of individual organs or tissue can be satisfied by using the sample in its entirety or subsampling to obtain the maximum weight required for the analysis. The tissue should be kept frozen during sub-sampling and weighing to prevent fluid migration or drainage.
- 11.4.1.11. Mercury analyses are performed according to Region IV method (nitric, sulfuric, and permanganate oxidation overnight, followed by reduction of excess permanganate with peroxide, and automated reduction and detection by vapor atomic absorption).
- 11.4.1.12. Digestion of tissue for multielement analyses is conducted according to the Region IV method (nitric peroxide) followed by ICP detection. Detection limit requirements are satisfied by manipulation of sample weight, final volume of digestate and in certain instances, detection by HGA furnace.
- 11.4.1.13. Other Tissue: Generally, other tissues will be prepared and analyzed the same as fish tissue with the additional precaution during preparation to observe closely and add additional reagents as required to thoroughly digest the sample.
- 11.4.1.14. EP/TCLP Extracts: Waste samples for EP Toxicity determinations will be extracted according to Method 1310 in SW-846 and subsequent clarifications of the methodology as received from Office of Solid Waste. The extract will be acid digested (nitric) (Method 3010 SW-846) and scanned by TCP for the usual twenty six elements. If any of the drinking water parameters are above the EP fail level (100 X drinking water MCL), the parameter(s) will be confirmed by furnace or PE 6000 as the element detection level may dictate.
- 11.4.1.15. Oil and Oily Samples: Oil emulsions and oil samples will be prepared on a wet weight basis. If the water phase can be separated, it is prepared as a water sample. The oil phase is weighed (1 g) into a small crucible and 2 mLs concentrated nitric acid added. The acid provides for dispersion of spike solutions and solubilization of particulates in the oil. The sample is heated at low heat on an oscillating hot plate until the acid is depleted. The crucible is transferred to a muffle furnace and brought up to 125°C for 1 hr. Increase temperature 175°C for 1 hr. Increase to 250°C for 1 hr. NOTE: Do not open furnace during the procedure and until furnace has cooled to 100°C. The sample is ashed overnight at 450°C maximum temperature. One mL of concentrated nitric acid and 1 mL of concentrated hydrochloric acid is added to the ash and warmed until ash is in solution. This solution is diluted to volume and is ready for analysis by ICP.

- 11.4.1.16. An alternate procedure for a total analysis of oil is the Parr bomb technique. A maximum of 0.2 g of oil can be digested which will raise the detection limits 5% over the dry ash method. Hg analyses can be conducted on these digestates.
- 11.4.1.17. High Volume Filters: Filter is quartered and one quarter is digested in 5 mLs aqua regia and 25 mLs water. Sample is heated on an oscillating hot plate at low heat until volume has been reduced to 10-15 mLs. Filter material is washed with DI water and the wash is made up to volume for analysis.
- 11.4.1.18. Special Samples: Samples received for analysis which are not amenable to the standard digestion techniques will be prepared according to the best judgement of the analyst. These cases will require additional documentation as to methodology, quality control, and justification of the method used.

#### 11.4.2. QC Requirements for Metals:

## 11.4.2.1. Sample Preparation

- 11.4.2.1.1. A blank solution will be prepared with each group of samples to monitor for contamination of reagents, glassware and laboratory.
- 11.4.2.1.2. A spike solution, prepared from standard reference materials (or laboratory standards that have been confirmed by SRM) will be prepared with each group of samples. (A group is defined as any batch of samples prepared together in the same hood at the same time and with the same reagents). This solution verifies instrument calibration and monitors the digestion procedure.
- 11.4.2.1.3. All projects will have at least one sample duplicated and spiked. Projects with large numbers of samples will be duplicated and spiked at the rate of ten percent.
- 11.4.2.2. Calibration Standards: Commercial single element standard solutions will be used for the preparation of instrument calibration solutions. These standards will be dated when received and their concentration verified with standard reference materials from NBS or reference samples from EMSL-Cincinnati, QA Branch. All commercial standards will undergo additional examination for trace contamination of elements other than the specified element. Mixes of these single element standards are prepared according to the requirements of the instrument being used.
- 11.4.2.3. Instrument Calibration: All instruments will be calibrated with working standards diluted from commercial stock solutions that have been verified to contain their stated concentration. Instruments will be calibrated to cover the range of concentrations found in the samples or the samples may be diluted to fall within the calibration range. An initial calibration check solution should agree within 2 percent.

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frequency that will validate all data generated for that set. Reference samples can also be considered as calibration check samples.

- 11.4.2.4. Instrument Log Books: Will be maintained to record all service and maintenance records.
- 11.4.2.5. Sample Analysis Records: Log books will be maintained to record preparation of samples to include record of duplicates, spikes, sample numbers, dates, analyst, etc.
- 11.4.2.6. Log books will be maintained at each instrument to record instrumental conditions and settings during the analysis of samples. (The JA 975 ICP log will include a copy of gains and profile setting for each set of samples.)
- 11.4.2.7. Data Records: All raw data from instrumentation will be retained for future reference.
- 11.4.2.8. QC Data: Data generated from sample duplicates, sample spikes, preparation blanks and SRM preparations will be compared with historical data for that particular sample type and, if found to be within acceptable limits, will be added to the QC data base. If the data are not within acceptable limits, sample will be reanalyzed. If, after a second analysis, the data still remains outside acceptable limits, data will be flagged and reported.
- 11.4.2.9. Glassware and Equipment: All glassware/teflon vessels will be placed into a "Micro" soak immediately after use and must not be allowed to dry while dirty. After thoroughly soaking, all detergent is removed by rinsing, followed by a 10% acid rinse and finally a thorough rinsing with DI water. Allow to drain on its side and seal with parafilm or a glass stopper before storage in an upright position. Pipets are rinsed immediately after use and placed in a "micro" soak until moved to an automatic rinser with DI water.

#### 11.5. General Inorganic

#### 11.5.1. Preparation and Analysis

11.5.1.1. Mitrogen, total Kjeldahl in Water - determinations are performed according to Method 351.2 MCAWW. This method requires manual digestion of the samples followed by automated colorimetric determination by Technicon AAII\. Calibration standards are digested with the samples to cover the expected concentration range. The range can be extended by dilution of the sample. The calibration curve will be checked for accuracy by analyzing a standard reference material sample with every group of samples. The calibration curve will be monitored throughout the run by analyzing a standard after every tenth (10th) sample to insure stability of calibration. Ten percent of the samples will be duplicated and spiked to monitor precision and accuracy of the procedure.

- 11.5.1.2. Mitrogen, Total Kjeldahl in Soil and Sediment determinations are performed on wet samples. A percent moisture determination is performed on a separate aliquot of the sample to facilitate conversion of results to a dry weight basis. Sample preparations are performed according to the Method 42, page 3-202 in Procedures for Handling and Chemical Analysis of Sediment and Water Samples. USEPA/Corp of Engineers. The distillate is quantitated for ammonia by Method 550.1, automated phenate, MCAAW, March 1983. The same criteria (i.e., TKN in water method) for verifying the calibration standards and generating precision/accuracy data apply.
- 11.5.1.3. Ammonia Mitrogen, water and wastes determinations are performed according to the distillation procedure, 417A 15th Ed., Standard Methods, 350.2 MCAWW, followed by the automated phenate method 350.1 MCAWW. The same QC requirements as stated under TKN in water and wastes apply.
- 11.5.1.4. Ammonia Hitrogum, soil and sediment determinations are performed according to the Region IV method of manual distillation followed by automated phenate quantitation by Method 350.1 MCAWW. This distillation procedure is referenced to Standard Methods for the Examination of Water and Wastewater, 15th Edition; MCAWW, 1983; and the Army Corps of Engineers 1976 manual, Ecological Evaluation of Proposed Discharge of Dredged and Fill Material into Navigable Waters. The same QC requirements as stated under TKN in water and wastes apply.
- 11.5.1.5. Mitrogen, Mitrate-Mitrite on water and wastes determinations are performed according to MCAWW, 1983 method 353.2, automated cadmium reduction. A calibration curve will be generated from standards made to cover the concentration range of the samples and confirmed with a standard reference material. Ten percent of the samples will be duplicated and spiked to indicate precision and accuracy for the run or group. After every tenth sample, a calibration standard will be analyzed to verify that the calibration curve has not changed.
- 11.5.1.6. Alkalinity determinations are performed according to method 310.1 MCAWW. A standard reference material sample is prepared and titrated with each batch of samples to verify accurate preparation and standardization of the 0.02N sulfuric acid solution. Precision is determined by analyzing samples in duplicate at a rate of 10%.
- 11.5.1.7. Acidity Samples are titrated to determine acidity according to Method 305.1 MCAWW. The 0.02N sodium hydroxide is standardized against 0.02N H₂SO₄. The Method 310.1 MCAWW. There is no standard reference solution for acidity; however, accuracy may be determined by spiking the sample with a calcium carbonate solution. Precision is determined by analyzing samples in duplicate at a rate of 10%.
- 11.5.1.8. Chloride Analysis of samples for chlorides are performed according to Method 325.3 MCAWW. The mercuric nitrate titrant is standardized against a standard sodium 12

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solution. A reference sample is titrated with each group of samples to verify accuracy of the method. A minimum of 10 percent of the samples are analysed in duplicate and 5 percent are spiked to assure precision and accuracy of the method.

- 11.5.1.9. COD Chemical Oxygen Demand of samples having a concentration greater than 50 mg/L are determined according to Method 410.1 MCAWW. When the concentration is less than 50mg/L, Method 410.2 is used. The ferrous ammonium sulfate titrant must be standardized daily against a standard potassium dichromate solution. A blank and a reference sample must be refluxed with each group of samples. Duplicates and spikes are run with each analysis to verify precision and accuracy. One sample from each group or set of samples will be duplicated and spiked to determine precision and accuracy of the method.
- 11.5.1.10. Color The platinum-cobalt colorimetric method is used to determine apparent color of water derived from naturally occurring materials (Method 110.2 MCAWW). Color is measured by visual comparison of the sample with platinumcobalt standards. The ADMI Method (Method 110.1 MCAWW) is used for color measurement on waters containing highly colored industrial wastes.
- 11.5.1.11. Conductance Conductivity is determined according to Method 120.1 MCAWW. A standard potassium chloride solution is used to check the accuracy of the conductivity meter. A reference sample and duplicates should be analyzed with each group of samples to determine precision and accuracy for the sample set.
- 11.5.1.12. Cyanide, Total, in Water Samples to be analyzed for total cyanide are distilled according to Method 335.2 MCAWW. After distillation, an automated pyridinebarbituric acid colorimetric method is used to determine quality of cyanide present. A blank, standard, and reference sample are distilled with each batch of samples. One sample in each sample batch must be duplicated and spiked to determine precision and accuracy.
- 11.5.1.13. Cyanide, Total in Soil and Sediment Approximately 10g of wet sample is used in this analysis. The cyanide is extracted from the sediment by stirring in a 10% sodium hydroxide solution for one hour. The procedure is described in Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue, EPA August 1977. After this pretreatment, the sediment and hydroxide solution are distilled and analyzed for cyanide as described above for total cyanide in water. A percent moisture determination is performed on a separate aliquot so results can be converted to a dry-weight basis. The same Q.C. requirements given above for total cyanide in water are applicable to cyanide in sediments also.

- 11.5.1.14. Cyanide, Amenable to Chlorination determinations are performed according to Method 335.1 MCAWW. Distillation and analysis are the same as for Total Cyanide in Water described above.
- 11.5.1.15. Fluoride, Total determinations require a preliminary distillation, Method 340.1 MCAWW. After distillation, fluoride concentration is determined using an Ion-Selective Electrode, Method 340.2. A reference sample is distilled and analyzed with each batch of samples to verify the calibration of a selective ion meter. Duplicates and spikes are analyzed to monitor precision and accuracy of the procedure.
- 11.5.1.16. Hardness determinations are performed according to Method 130.2 MCAWW. The 0.02M EDTA titrant is standardized against a standard calcium carbonate solution. A reference sample is analyzed with each batch of samples to verify accuracy of method.
- 11.5.1.17. Phenolics, Total Recoverable the automated 4-aminoantipyrine (4-AAP) colorimetric procedure, Method 420.2 MCAWW, is used following sample distillation. A blank, a standard, and a reference sample are distilled and analyzed with each batch of samples to verify accuracy of method. Samples will be analyzed in duplicate at a rate of 10% to determine precision of analysis.
- 11.5.1.18. Phosphorus, all forms determinations are performed according to Method 365.1 MCAWW. Total phosphorus requires a manual digestion of the samples followed by automated colorimetric determinations using a Technicon AAII. A series of standards from 0 to 2.0 mg/l, total phosphorus, a reference sample, and a blank are digested with each batch of samples. Samples to be analyzed for ortho-phosphate are filtered in the field, and run on the Technicon AAII without prior digestion. Ten percent (10%) of the samples are duplicated, and five percent (5%) are spiked to monitor precision and accuracy of the method.
- 11.5.1.19. Phosphorus, Total in Soil and Sediment determinations are performed on 0.1 to 0.2g of dry sample. Sample preparations are performed according to the Region IV method (Region IV LSB Methods for the Analysis of Sediments and Sludges March 1981). After manual digestion, phosphorus is determined by the automated colorimetric procedure, Method 365.1 MCAWW on the Technicon AAII. A series of standards and a reference sample are digested with each batch of samples. Duplicates and spikes are analyzed to describe precision and accuracy of the sample set.

11.5.1.20. Sulfate - determinations are performed by a turbidimetric method, Method 375.4 MCAWW. The working concentration range is from 1 to 40 mg SO₄/L, but this range can be extended by dilution of samples. The sulfate standards are prepared from standard D.lN sulfuric acid solution or from a standard NaSO₄ solution. The sulfuric acid solution must be standardized against a sodium carbonate solution. A reference sample is analyzed with each batch of samples to verify accuracy of calibration standards. Samples are analyzed in duplicate at a rate of 10% to determine precision of analysis.

- 11.5.1.21. Organic Carbon, Total determinations are performed according to Method 415.1 MCAWW. Organic carbon is converted to carbon dioxide by catalytic combustion and is measured directly by an infrared detector using a Beckman 915B Total Organic Carbon Analyzer. A series of standards from 1 to 100 mg carbon/L made from a potassium hydrogen phthalate stock solution are used to prepare a calibration curve. The accuracy of the calibration curve is verified by analyzing a standard reference sample. Duplicates and spikes are analyzed to describe precision a the sample set.
- 11.5.1.22. Turbidity determinations are made according to method 180.1 MCAWW. A standard reference solution is analyzed with each batch of samples to verify proper calibration of the turbidimeter.
- 11.5.1.23. Biochemical Oxygen Demand (5 day, 20°C) determinations are made according to the procedure found in Standard Methods for the Examination of Water and Wastewater, 15th Edition, p. 483, Method 507 (1980). A standard reference sample is analyzed with each group of samples to verify accuracy of the procedure. If soluble BOD is required, the sample is filtered through a glass fiber filter prior to analysis by the above method. Samples are analyzed in duplicate at a rate of 10% to determine precision of the analysis.
- 11.5.1.24. Oil and Grease, Total Recoverable are analyzed according to Method 413.1 MCAWW. A blank and a reference sample are extracted with each group of samples.
- 11.5.1.25. pH determinations are made according to Method 150.1 MCAWW. Two buffer solutions are used to calibrate the pH meter. A Standard reference solution is available to verify accuracy of commercial buffer solutions used for calibration.
- 11.5.1.26. Residue, Filterable (dried at 180°C) determinations are made according to Method 160.1 MCAWW. A standard reference solution is analyzed with each group of samples to verify accuracy

of procedure. Samples are analyzed in duplicate at a rate of 10% to determine precision of the analysis.

- 11.5.1.27. Residue, Non-Filterable dried at 103-105°C total suspended solids or non-filterable residue are analyzed according to Method 160.2 MCAWW. A reference sample is analyzed with each batch of samples to verify accuracy of the procedure. Samples are analyzed in duplicate at a rate of 10% to determine precision of the analysis.
- 11.5.1.28. Residue, Total, Volatile, and Settleable Matter determinations are made according to Methods 160.3, 160.4, 160.5 MCAWW, respectively. Samples are duplicated to monitor precision of procedure.
- 11.5.1.29. Sulfide determinations are made according to a procedure given in the <u>Chemistry Laboratory Manual *Bottom Sediments*</u> compiled by Great Lakes Region Committee on Analytical Methods, EPA-FRQA, Dec. 1969. The methylene blue color formed in the samples and standards is measured with a spectrophotometer at 650 nm. Duplicates and spikes are analyzed with each batch of samples to monitor the precision and accuracy of the method.
- 11.5.1.30. Beat of Combustion determinations are made according to ASTM Method D2382-76 (1980) "Standard Test Method for Heat of Combustion of Hydrocarbon Fuels by Bomb Calorimeter (High Precision Method)." Analyses are performed on a Parr 1241 Adiabatic Oxygen Bomb Calorimeter with a 1720 procedure. A standard benzoic acid pellet is analyzed with each batch of samples to verify calibration of the calorimeter.

#### 11.6. OC Requirements for General Inorganic

- 11.6.1. Data Records: All raw data from instrumentation will be retained for future reference. Where readings are read directly from an instrument, these readings are considered raw data and are recorded in the appropriate log book.
- 11.6.2. QC Data: Data generated from sample duplicates, spikes, blanks and SRM preparations will be compared with historical data from that particular sample type for acceptability. If the data are within the limits of the test, they are added to the QC data base. If it is not within the limits, the sample/samples will be reanalyzed. If, after the second analysis, the data still does not fall within the acceptance limits, the data will be flagged, reported and the QC data is not added to the data base. Certain analyses such as BOD, that cannot be repeated, will be reported with flag if QC data indicates the test to be outside acceptance limits.

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#### 12. MICROBIOLOGICAL QUALITY CONTROL DATA

12.1. The quality control checks listed below are routinely performed. The resulting data are recorded on statistical worksheets (Form 12-1) and/or in the Microbiology Quality Control Log.

#### 12.1.1. Quantitative Data (MF-MPW Analyses)

- 12.1.1.1. Every 10th sample is duplicated except in the case of NPDES Compliance Monitoring.
- 12.1.1.2. If MPDES Compliance Monitoring is being conducted, a minimum of one sample per effluent is replicated three times.
- 12.1.1.3. A start and finish MF control test (rinse water, medium, and supplies must be conducted for each filtration series. If sterile controls indicate contamination, all data on samples affected must be rejected.
- 12.1.1.4. Public water supply samples analyzed by the membrane-filter method must have at least five sheen or borderline colonies verified from each membrane containing five or more such colonies. Counts must be adjusted based on verification.
- 12.1.1.5. The MPN test must be carried to completion, except for gram staining, on 10 percent of the positive confirmed public water supply samples. Counts must be adjusted based on completion results.
- 12.1.1.6. Periodically, MPN tube code results are tabulated to check the frequency of occurrence of various codes. Observed code frequencies are compared to theoretical expected frequencies.

#### 12.1.2. Qualitative Data

12.1.2.1. Accuracy of biochemical and serological isolate identifications is checked by submitting cultures to the National Center for Disease control (Atlanta) for confirmation.

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# **FORMS**

## MICROBIOLOGICAL QUALITY CONTROL DATA

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## STATISTICAL WORKSHEET

			<u> </u>						
Log NoSampleEnumeration Method: Bacterial Indicator: Description		Date	A	nalyst_		x	_ <u>x</u>		
		MF MPN Total Coliform		Or Pr	ther_	x	x _b		
( denotes									
Rep.	Xa	Xa2	- Xb	Xb2	Хc	Xc2	<b>X</b> d	Xd2	
1									
2									
3									
4	1								
5								<u> </u>	
 5								<del>                                     </del>	
7		-							
8									
9								<del></del>	
10						<del> </del> "			
$EX_a = (EX_a)^2 =$	$\frac{EX_{b}}{(EX_{b})^{2}} =$		$EX_{c} = (EX_{c})^{2} =$		EX _d = (EX _d ) ² =				
<b>X</b> a =	<b>T</b> _b =		I =		<b>x</b> _a =				
ex _a ² =	EX _b ²	=	EX _c ²		EXd	² =			
n ex _a =	n EX	_b ² =	n EX	,² <b>-</b>	n E	$x_d^2 =$			
s = <u>+</u> RSD = %	e = RSD	<u> </u>	s = + RSD = %		g = <u>+</u> RSD = %				

## EQUATIONS

$$n = \pm \sqrt{\frac{nEX^2 - (EX)^2}{n(n-1)}}$$
RSD % = s X 100

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#### 13. PERFORMANCE QUALITY CONTROL DATA HANDLING

13.1. All performance quality control data (Section 10, 11, and 12) are transferred from the data books and forms to the appropriate quality control logs or data entry forms. Quality control logs or forms are maintained for inorganic parameters, organics and pesticides, metals and microbiological parameters.

13.2. The following subsections contain the techniques used to measure analytical performance:

#### 13.2.1. Precision Data

13.2.1.1. Precision is expressed as percent relative standard deviation and is calculated by the formula:

Where:  $\underline{S}$  = Standard Deviation  $\underline{X}$  = Mean

13.2.2. The estimated standard deviation may be calculated by the following equations fpr duplicate analysis:

$$S = \pm (X_1 - X_2) 0.89$$

Where:  $X_1$  and  $X_2$  = individual observations.

For replicate analysis (any number >2)

$$S = \frac{ +n E X^{2} - (E X)^{2}}{n(n-1)}$$

NOTE: Automatic calculators
may be used to deter
mine S if this formula

is used.

Where: X = individual observations.

n = number of observations.

Do not use this formula for n=2.

#### 13.3. Accuracy Data

13.3.1. Accuracy is expressed as percent recovery and calculated by the formula:

Where: X = concentration in unspiked sample.

2 = concentration in spiked sample.

T - True concentration of spike added.

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## 13.4. Annual Analytical Performance Summary

13.4.1. At the end of each fiscal year, a summary report of the Branch's analytical performance is prepared. Contained in this report are: the precision data (average percent RSD, upper warning and control limits), and accuracy data (average total percent recovery of spiked samples, AQC reference samples, and performance audit samples). This summary will contain all parameters for which adequate quality control data have been generated during the year. Special summaries of the Branch's QC data can be made at any time using SAAMS.

## 13.4.2. Participation in EPA Performance Evaluation Studies.

13.4.3. The Branch will participate in announced EPA performance Evaluation Studies. Performance on these studies further indicates the effectiveness of the laboratory's day-to-day quality control procedure.

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## 14. QUALITY ASSURANCE OVERVIEW OF CONTRACTOR DATA

14.1. The Branch has the responsibility for providing QA/QC overview of the contractor-generated laboratory data submitted to the Division. Currently, the principle source of contractor data overviewed by the Branch is generated by the Contract Laboratory Program (CLP). The Branch is involved in several facets of CLP operations and data overview including:

- 14.2. Technical Project Officer (TPO) activities. A member of the staff (Tom Bennett) serves as the Region IV CLP TPO. The TPO works directly with Region IV CLP laboratories on contract, technical, capacity, timeliness, and data quality issues.
- 14.3. CLP Technical Caucuses. Staff members serve on technical caucuses responsible for developing and reviewing analytical and data review protocols aimed at improving the CLP.
- 14.4. Data Review. The detailed technical review of laboratory data constitutes the major activity associated with contractor quality assurance overview. Data reviews are conducted by either ASB or Environmental Services Assistance Team (ESAT) personnel. All ESAT reviews are overviewed by EPA personnel. The data review activities consist of the following elements:
  - 14.4.1. Blind blank and spike samples are prepared and submitted to the contractors with each set of samples. Contractor analysis of these samples is evaluated as part of the final data review. Duplicates of the blanks and spikes are analyzed by our Branch laboratory for comparative purposes.
  - 14.4.2. When CLP services are requested, each case, its' associated sample numbers, and the type of analyses requested are logged into the computerized data management system known as the Sampling and Analysis Management System (SAAMS). The Branch secretary has the responsibility of tracking all CLP cases. The SAAMS facilitates the tracking process by allowing the secretary to determine when the cases are due and when all samples from a specific case have been received.
  - 14.4.3. Once the completed case is received, the data package (all deliverables as specified in the CLP Statement of Work) is sent to ASB or contractor support personnel (ESAT) for technical review. EPA and contractor personnel use <u>Laboratory Data Validation Functional</u>

    <u>Guidelines for Evaluating Organic and Inorqanic Analyses</u>, which was prepared by U.S. EPA Headquarters and the EPA Regions, as the basis for their Routine Analytical Services(RAS) technical data review. In addition to the Functional Guidelines, the Region IV Standard Operating Procedure (SOP) for Organic and Inorganic data review (October 1, 1987) is used. The regional SOP supplements the national Functional Guidelines in areas where the national protocol is incomplete or unclear. The regional SOP also contains specific

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instructions for arranging the numerical data and data qualifiers in a format compatible with SAAMS.

14.4.4. EPA and/or ESAT personnel validate 100t of the data received from the CLP. All data packages are thoroughly reviewed for sample holding times. instrument performance, instrument calibration, blind QC samples, blanks, surrogate recoveries, matrix spike recoveries, qualitative identifications, and an overall case assessment is made. When technical deficiencies are noted within a case, the individual data items affected are assigned a data qualifier. If no technical problems are discovered, the data items will be left unflagged. Data items with qualifiers inform the data user that some problem(s) exists with the quantitative or qualitative accuracy of the data. The data qualifiers used in Region IV are primarily those listed in the national Functional Guidelines; however, there are a few instances where region specific qualifiers are used. These region specific qualifiers and their application are explained in the Region IV SOP for data review.

14.4.5. All CLP data, i.e., the validated results of the analyses, are entered in to the SAAMS. The SAAMS is able to accommodate both the numerical value and any data qualifiers used. Unvalidated CLP data is electronically transferred from Sample Management Office (SMO) to the RTP mainframe and the Region IV SAAMS from the diskette provided with each case by the contract laboratory. If the transfer is timely and data arrive before the regional data validation process is completed, the electronically transferred data is edited and the necessary data qualifiers are added. If the data are not electronically transferred from SMO before the data validation process is completed, the validated data must be manually entered into the SAAMS.

14.4.6. The data review documentation assessing the data quality of each case is distributed to the appropriate regional Technical Project Officer, the EPA Headquarters Analytical Operations Branch, and EMSL - Las Vegas. The validated analytical data for each case is distributed to the site project manager, the data requestor, and a copy is retained for Region IV files. In addition to the file copy, the data is also retained by the region on computer tape via the SAAMS system.

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## 15. AMALYTICAL CORRECTIVE ACTIONS

15.1. Corrective action will be taken at any time during the analytical process when deemed necessary based on analyst judgement or when quality control data indicate a need for action. Generally, corrective action will be triggered by such things as: poor analysis replication, poor recovery, instrument calibration problems, blank contamination, etc. (See previous sections for specifics)

- 15.2. Corrective actions will include, but not necessarily be limited to: reanalysis, calculation checks, instrument recalibration, preparation of new standards/blanks, re-extraction/digestion, dilution, application of another analysis method, additional analysts training, etc. Most frequently, these corrective actions will be initiated by the analyst at the time of analysis. However, some corrective actions are initiated subsequent to analysis based on evaluations performed by quality assurance or laboratory management personnel.
- 15.3. All data corrective actions will be noted on the appropriate log, chromatogram, strip chart or data report.

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#### 16. DATA QUALITY OBJECTIVES

16.1. During the planning phase of a project requiring laboratory support, the data user must establish the quality of data required from the investigation. Such statements of data quality are known as Data Quality Objectives (DQO's). The DQO's are qualitative and quantitative statements of the quality of data required to support specific decisions or regulatory actions. The laboratory is responsible for producing data of known quality and consistent with that prescribed in the DQO.

- 16.2. The laboratory will select analytical methods, instruments, parameter detection limits, etc. which are capable of producing data of the quality required by the DQO. The quality of a data set is defined in terms of: precision, accuracy, representativeness, completeness and comparability. The significance of each of these measures differs according to their applicability to the laboratory and to a particular data set. A brief explanation of the above measures are as follows:
  - 16.2.1. Precision and accuracy. These are quantitative measures that characterize the amount of variability and bias inherent in a given data set. Precision refers to the level of agreement among repeated measurements of the same characteristic. Accuracy refers to the difference between an estimate based on the data and the true value of the parameter being estimated (See Section 13).
  - 16.2.2. Representativeness. Refers to the degree to which the data collected accurately reflect the population, group or medium being sampled.
  - 16.2.3. Completeness. Refers to the amount of data that is successfully collected with respect to that amount intended in the study design.
  - 16.2.4. Comparability. Refers to the ability to compare data from different sources with a degree of confidence.

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#### 17. LABORATORY EVALUATIONS

#### 17.1. <u>Introduction</u>

17.1.1. The Analytical Support Branch has the responsibility for laboratory evaluations in support of the EPA national and regional quality assurance programs. These laboratories include:

- o state principal drinking water laboratories for the purpose of Interim Certification under the Safe Drinking Water Act.
- o state water quality and hazardous waste laboratories.
- o municipal and industrial laboratories as part of the DMR QA and Performance Audit Inspection programs, CLP laboratories located in Region IV, and laboratories providing support to Superfund removal and remedial programs.
- 17.1.2. This laboratory overview is conducted by on-site evaluations of laboratories and the coordination of national performance audit studies. Laboratory evaluation will be consistent with the Region IV State Overview policy.

## 17.2. Standard Operating Procedure for State On-site Evaluations

#### 17.2.1. Pre-evaluation Planning

- 17.2.1.1. The state agency will be notified in writing at least thirty days prior to an on-site visit. Telephone contacts with laboratories may be made prior to the formal notification for the purpose of setting convenient dates for the evaluations.
- 17.2.1.2. Pre-survey questionnaires will be sent to the state laboratories in time for completion prior to the on-site visit. Where available, questionnaires given in Agency criteria and procedures documents will be used.
- 17.2.1.3. The notification and scheduling of Performance Audit Inspections will be done by the Environmental Compliance Branch.

## 17.2.2. Scope of On-Site Evaluations

#### 17.2.2.1. Personnel

17.2.2.1.1. A determination is made as to the adequacy of laboratory staff's size, training, and experience for the performance of its function.

#### 17.2.2.2. Facilities

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17.2.2.2.1. An inspection of facilities is conducted to determine the adequacy of laboratory space, sample storage space, office space and services. Safety issues are also addressed.

## 17.2.2.3. Equipment

17.2.2.3.1. Equipment and instrumentation are examined to determine if they are adequate and meet the specifications for analyses being performed. Preventive maintenance logs are examined.

#### 17.2.2.4. Sample Handling

17.2.2.4.1. Sample handling procedures are reviewed to determine if EPA recommendations for containers, preservation, and holding times are being adhered to. Sample identification, traceability, and chain-of-custody procedures are examined.

## 17.2.2.5. Methodology

17.2.2.5.1. The methods in use at the laboratory are examined to determine if they are EPA approved test procedures (or approved alternate procedures) as outlined in the appropriate regulation(s).

#### 17.2.2.6. Data Handling

17.2.2.6.1. Data handling procedures are reviewed for adequacy of raw data documentation, data validation, and reporting.

## 17.2.2.7. Quality Assurance

17.2.2.7.1. A review is made of the laboratory's quality assurance activities to determine:

17.2.2.7.1.1. If SOP's have been developed and are in use.

17.2.2.7.1.2. If an adequate amount of performance quality control data are being generated and how these data are used,

#### 17.2.3. Exit Interview

17.2.3.1. Following the evaluation, an exit interview with the laboratory director and staff will be conducted by the evaluation team. This interview will summarize the findings and recommendations that will be covered in the evaluation report.

#### 17.2.4. Evaluation Report

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17.2.4.1. A final report will be completed within thirty days of the evaluation visit. The report will be written in the standard Region IV format.

## 17.3. Performance Audits

17.3.1. An important element of the evaluation program is the participation of laboratories in Agency performance audits. These audits involve the analysis of unknown performance evaluation samples.

17.3.2. Laboratory participation and performance will be tracked to ensure that regulatory and Region IV policy requirements are met.

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